

STIC Search Report

Biotech-Chem Library

STIC Database Tracking Number: 115545

TO: Shailendra Kumar
Location: Rem 5d61 / 5c18
Monday, March 08, 2004
Art Unit: 1621
Phone: 272-0640
Serial Number: 09 / 774232

From: Jan Delaval
Location: Biotech-Chem Library
Rem 1A51
Phone: 272-2504

jan.delaval@uspto.gov

Search Notes

Jan please

114902

Access DB# 115545

SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: S. Kumar Examiner #: 69594 Date: 2/23/04
Art Unit: 162 Phone Number 301-272-0146 Serial Number: 09/774232
Mail Box and Bldg/Room Location: REM 5DE1 Results Format Preferred (circle): PAPER DISK E-MAIL
5C70

If more than one search is submitted, please prioritize searches in order of need.

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Material Processing by repeated solvent expansion-contraction
Inventors (please provide full names): Said Savim et al.

Earliest Priority Filing Date: 3/3/2000

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

(The solute is acetaminophen)

- A method for producing solute comprising
- a) dissolving solute in a liquid solvent to form solvent/solute phase
 - b) dissolving gaseous fluid in the solvent/solute liquid phase
 - c) causing solvent/solute/gaseous liquid phase to expand
 - d) causing gaseous fluid to be dissolved to a concentration such that the solvent/solute/gaseous fluid liquid phase expands until it loses its affinity for solubilization of said solute and said solute precipitates
 - e) retaining precipitated solute on a retention medium
 - f) reducing the pressure in the liquid phase to a point where a
 - g) optionally adding more solute to the liquid phase.

See claims 1-11 + 22-66

STAFF USE ONLY

	Type of Search	Vendors and cost where applicable
Searcher: <u>Cam</u>	NA Sequence (#) _____	STN <input checked="" type="checkbox"/>
Searcher Phone #: <u>72504</u>	AA Sequence (#) _____	Dialog _____
Searcher Location: _____	Structure (#) _____	Questel/Orbit _____
Date Searcher Picked Up: <u>3/8</u>	Bibliographic <input checked="" type="checkbox"/>	Dr. Link <u>1015</u>
Date Completed: <u>3/8</u>	Litigation _____	Lexis/Nexis _____
Searcher Prep & Review Time: _____	Fulltext _____	Sequence Systems <u>26 6000</u>
Clerical Prep Time: <u>15</u>	Patent Family _____	WWW/Internet <u>6000</u>
Online Time: <u>+100</u>	Other _____	Other (specify) _____

=> fil reg

FILE 'REGISTRY' ENTERED AT 14:51:01 ON 08 MAR 2004
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
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Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES: 7 MAR 2004 HIGHEST RN 659718-58-8
DICTIONARY FILE UPDATES: 7 MAR 2004 HIGHEST RN 659718-58-8

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2004

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more
information enter HELP PROP at an arrow prompt in the file or refer
to the file summary sheet on the web at:
<http://www.cas.org/ONLINE/DBSS/registryss.html>

=> d ll ide can

L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN

RN 124-38-9 REGISTRY

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

OTHER NAMES:

CN Carbon oxide (CO2)

CN Carbon-12 dioxide

CN Carbon-12C dioxide-1602

CN Carbonic acid anhydride

CN Carbonic acid gas

CN Carbonic anhydride

CN Dry ice

CN Khladon 744

CN R 744

FS 3D CONCORD

DR 18923-20-1

MF C 02

CI COM

LC STN Files: ADISNEWS, AGRICOLA, ANABSTR, AQUIRE, BIOBUSINESS, BIOSIS,
BIOTECHNO, CA, CABA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CBNB, CEN,
CHEMCATS, CHEMINFORMRX, CHEMLIST, CHEMSAFE, CIN, CSCHM, CSNB, DDFU,
DETERM*, DIOGENES, DIPPR*, DRUGU, EMBASE, ENCOMPLIT, ENCOMPLIT2,
ENCOMPPAT, ENCOMPPAT2, GMELIN*, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB,
IPA, MEDLINE, MRCK*, MSDS-OHS, NIOSHTIC, PDLCOM*, PIRA, PROMT, RTECS*,
SPECINFO, TOXCENTER, TULSA, ULIDAT, USAN, USPAT2, USPATFULL, VETU, VTB
(*File contains numerically searchable property data)

Other Sources: DSL**, EINECS**, TSCA**

(**Enter CHEMLIST File for up-to-date regulatory information)

O=C=O

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

174398 REFERENCES IN FILE CA (1907 TO DATE)

669 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA

174609 REFERENCES IN FILE CAPLUS (1907 TO DATE)
21 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:173564
REFERENCE 2: 140:173485
REFERENCE 3: 140:173471
REFERENCE 4: 140:173329
REFERENCE 5: 140:173266
REFERENCE 6: 140:173203
REFERENCE 7: 140:172374
REFERENCE 8: 140:172051
REFERENCE 9: 140:172028
REFERENCE 10: 140:172024

=> d his

(FILE 'HOME' ENTERED AT 13:39:27 ON 08 MAR 2004)
SET COST OFF

FILE 'REGISTRY' ENTERED AT 13:39:36 ON 08 MAR 2004
L1 1 S CARBON DIOXIDE/CN

FILE 'HCAPLUS' ENTERED AT 13:39:44 ON 08 MAR 2004
L2 174782 S L1
L3 438211 S CO2 OR CARBON() (DIOXIDE OR DI OXIDE)
L4 443621 S L2,L3
E SOLUTE/CT
L5 4846 S E20-E23
E E20+ALL
L6 4846 S E2
L7 1962 S DISSOLVED SUBSTANCE
L8 78132 S E4-E27
L9 6713 S L5-L7
L10 1980 S SOLVENT AND L9
E SOLVENT/CT
L11 46150 S E53-E85
E E53+ALL
L12 48190 S E2+NT
E E18+ALL
L13 45384 S E2,E1+NT
L14 1062 S L9 AND L11-L13
L15 1133 S L8 AND L11-L13
L16 7863 S L8 AND SOLVENT
L17 9795 S L10,L14-L16
L18 512 S L17 AND (GAS OR GASEOUS)
E GAS/CT
L19 0 S L17 AND E3
E GASES/CT
L20 131 S L17 AND E3+NT
L21 20 S L17 AND E3-E25
E E3+ALL
L22 0 S L17 AND E5

L23 188 S L17 AND L4
 L24 703 S L18, L20, L21, L23
 L25 25 S L24 AND (EXPAND? OR EXPANSION?)
 L26 0 S L24 AND RETRACT?
 L27 3 S L24 AND CONTRACT?
 L28 22 S L24 AND PRECIPITAT?
 L29 48 S L24 AND EXTRACT?
 L30 36 S L24 AND COAT?
 L31 23 S L24 AND RETENTION
 L32 11 S L24 AND ?FILTR?
 L33 16 S L24 AND ?FILTER?
 L34 34 S L24 AND ?CRYS?
 L35 170 S L25-L34
 L36 8 S L35 AND LIQUID PHASE
 E SAIM S/AU
 L37 27 S E3, E4
 E HORHOTA S/AU
 L38 15 S E4-E8
 E BOCHNIAK D/AU
 L39 5 S E4-E6
 E BOEHRING/PA, CS
 L40 8232 S E4-E9 OR BOEHRINGER?/PA, CS
 E BOHRINGER/PA, CS
 L41 8 S E3-E9
 E BORINGER/PA, CS
 E BOEINGER/PA, CS
 E BOERINGER/PA, CS
 L42 10 S L37-L39 AND L40, L41
 L43 1 S L42 AND L35
 L44 78193 S L9 OR SOLUTE
 L45 154348 S L8, L44
 L46 28860 S L45 AND ?SOLVENT?
 L47 5220 S L45 AND L11-L13
 L48 28922 S L17, L46, L47
 L49 2675 S L48 AND (GAS OR GASEOUS)
 L50 466 S L48 AND GASES+NT/CT
 L51 988 S L48 AND L4
 L52 3539 S L24, L49-L51
 L53 123 S L52 AND (EXPAND? OR EXPANSION?)
 L54 6 S L52 AND (RETRACT? OR CONTRACT?)
 L55 114 S L52 AND PRECIPITAT?
 L56 341 S L52 AND EXTRACT?
 L57 152 S L52 AND COAT?
 L58 362 S L52 AND RETENT?
 L59 64 S L52 AND (?FILTR? OR ?FILTER?)
 L60 224 S L52 AND ?CRYS?
 L61 3 S L54 AND L53, L55-L60
 L62 2 S L61 NOT LITHIUM/TI
 L63 2 S L37-L39 AND L52
 L64 1 S L42 AND L52
 L65 3 S L43, L62-L64
 L66 9 S L42 NOT L65
 SEL DN AN 1-6
 L67 6 S L66 AND E1-E18
 L68 9 S L65, L67 AND L2-L67
 L69 594 S L48 AND (SUPERCRITIC? OR SUPER CRITIC?) () FLUID?
 L70 45 S L69 AND (EXPAND? OR EXPANSION?)
 L71 0 S L70 AND (RETRACT? OR CONTRACT?)
 L72 26 S L70 AND (EXTRACT? OR COAT? OR RETENT? OR ?CRYS?)
 L73 915 S L48 AND (SUPERCRITIC? OR SUPER CRITIC?)
 L74 3808 S L73, L52
 L75 142 S L74 AND (EXPAND? OR EXPANSION?)
 L76 9 S L75 AND (CONTRACT? OR RETRACT? OR RETENTION?)

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                SEL DN AN 2
L77             1 S E19-E21
L78             9 S L68,L77
L79            139 S L75 NOT L78
L80             63 S L79 AND (?CRYS? OR PRECIPITAT? OR EXTRACT? OR COAT? OR ?FILTR
L81             72 S L78,L80
L82             30 S L37-L39 NOT L81
                SEL DN AN 2
L83             1 S E22-E24
L84            73 S L81,L83
L85            64 S L84 AND (?SOLUTE? AND ?SOLVENT?)
L86            56 S L85 AND (GAS OR GASEOUS OR L4)
L87            45 S L85 AND (SUPERCritical? OR SUPER CRITICAL?)
L88            64 S L86,L87
L89             9 S L84 NOT L88
                SEL DN AN 3 9
L90             7 S L89 NOT E25-E30
L91            71 S L88,L90
L92            64 S L91 AND EXPAN?
L93            20 S L92 AND (RETRACT? OR CONTRACT? OR RETENTION? OR EXTRACT?)
L94            44 S L92 NOT L93
L95            27 S L90,L93
L96            44 S L91 NOT L95
L97            44 S L94,L96
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FILE 'REGISTRY' ENTERED AT 14:49:58 ON 08 MAR 2004

FILE 'HCAPLUS' ENTERED AT 14:50:04 ON 08 MAR 2004

L98 27 S L95 AND L2-L97

FILE 'REGISTRY' ENTERED AT 14:51:01 ON 08 MAR 2004

=> fil hcaplus

FILE 'HCAPLUS' ENTERED AT 14:51:05 ON 08 MAR 2004

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FILE COVERS 1907 - 8 Mar 2004 VOL 140 ISS 11

FILE LAST UPDATED: 5 Mar 2004 (20040305/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d 198 all hitstr tot

L98 ANSWER 1 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:875319 HCAPLUS

DN 139:354439

ED Entered STN: 07 Nov 2003

TI Method for reduction of residual organic solvent in carbomer

IN Forness, Cecile; Horhota, Stephen T.; Saim, Said;
Bochniak, David
PA Boehringer Ingelheim Pharmaceuticals, Inc., USA
SO PCT Int. Appl., 24 pp.
CODEN: PIXXD2
DT Patent
LA English
IC ICM C08F006-00
ICS C08F006-28; B01D011-02; A61K009-00
CC 63-5 (Pharmaceuticals)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2003091290	A1	20031106	WO 2003-US12403	20030421
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
	US 2003211159	A1	20031113	US 2003-419436	20030421
PRAI	US 2002-374919P	P	20020423		
AB	The method with effective for the reduction of residual organic solvent in carbomer down to the ppm level (e.g., ≤ 30 ppm), comprises exposing a carbomer (Carbomer 934P) containing residual organic solvent (e.g., benzene) to a gaseous fluid (e.g., CO ₂) in which the residual organic solvent is soluble and under conditions sufficient to extract at least some of the residual organic solvent from the carbomer. A pharmaceutical suspensions contain the carbomers treated by the method and a therapeutically active agent.				
ST	carbomer residual org solvent redn				
IT	Vasodilators (diuretic, therapeutically active agent; method for reduction of residual organic solvent in carbomer)				
IT	Drugs (method for reduction of residual organic solvent in carbomer)				
IT	Analgesics Anesthetics Anti-inflammatory agents Antibiotics Anticoagulants Antihistamines Antimicrobial agents Antioxidants Antipsychotics Antitumor agents Antiviral agents Decongestants Fungicides Hypnotics and Sedatives Immunosuppressants Nervous system stimulants Thrombolytics (therapeutically active agent; method for reduction of residual organic solvent in carbomer)				
IT	Amino acids, biological studies Hormones, animal, biological studies				

Minerals, biological studies
 Neurotransmitters
 Nucleotides, biological studies
 Peptides, biological studies
 Proteins
 Vitamins

RL: BUU (Biological use, unclassified); BIOL (Biological study); USES
 (Uses)

(therapeutically active agent; method for reduction of residual organic solvent in carbomer)

IT 57916-92-4, Carbomer 934P

RL: BUU (Biological use, unclassified); BIOL (Biological study); USES
 (Uses)

(method for reduction of residual organic solvent in carbomer)

IT 79-10-7DP, Acrylic acid, polymers 9003-97-8P, Polycarbophil
 9007-16-3P, Carbomer 934 9062-04-8P, Carbomer 941 76050-42-5P,
 Carbomer 940 96827-24-6P, Carbomer 1342 126040-58-2P, Calcium
 polycarbophil

RL: BUU (Biological use, unclassified); PUR (Purification or recovery);
 BIOL (Biological study); PREP (Preparation); USES (Uses)

(method for reduction of residual organic solvent in carbomer)

IT 71-23-8, Propanol, uses 71-36-3, Butanol, uses 74-84-0, Ethane, uses
 74-85-1, Ethylene, uses 74-98-6, Propane, uses 75-28-5, Isobutane
 75-46-7, Trifluoromethane 75-73-0, Tetrafluoromethane 95-47-6,
 o-Xylene, uses 106-97-8, Butane, uses 110-82-7, Cyclohexane, uses
 115-07-1, Propylene, uses 115-11-7, Isobutene, uses **124-38-9,**
Carbon dioxide, uses 2551-62-4, Sulfur hexafluoride
 7664-41-7, Ammonia, uses 7732-18-5, Water, uses 10024-97-2, Nitrous
 oxide, uses

RL: NUU (Other use, unclassified); USES (Uses)

(method for reduction of residual organic solvent in carbomer)

IT 56-23-5, Carbon tetrachloride, processes 64-17-5, Ethanol, processes
 67-56-1, Methanol, processes 67-63-0, Isopropanol, processes 67-64-1,
 Acetone, processes 67-66-3, Chloroform, processes 67-68-5, Dimethyl
 sulfoxide, processes 71-43-2, Benzene, processes 75-09-2, Methylene
 chloride, processes 75-35-4, 1,1-Dichloroethene, processes 79-01-6,
 Trichloroethylene, processes 107-06-2, 1,2-Dichloroethane, processes
 108-88-3, Toluene, processes 108-95-2, Phenol, processes 110-54-3,
 Hexane, processes 123-91-1, 1,4-Dioxane, processes 141-78-6, Ethyl
 acetate, processes

RL: REM (Removal or disposal); PROC (Process)

(residual; method for reduction of residual organic solvent in carbomer)

IT 5534-09-8, Beclomethasone dipropionate 9004-10-8, Insulin, biological
 studies 13392-18-2, Fenoterol 18559-94-9, Albuterol 22254-24-6,
 Ipratropium bromide 30286-75-0, Oxytropium bromide 37148-27-9,
 Clenbuterol 51022-70-9, Albuterol sulfate 71125-38-7, Meloxicam
 136310-93-5, Tiotropium bromide

RL: BUU (Biological use, unclassified); BIOL (Biological study); USES
 (Uses)

(therapeutically active agent; method for reduction of residual organic solvent in carbomer)

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Basf Ag; WO 9720866 A 1997 HCAPLUS
- (2) Boehringer Ingelheim Pharma; WO 9909990 A 1999 HCAPLUS
- (3) Bresciani, A; US 5093472 A 1992 HCAPLUS

IT **124-38-9, Carbon dioxide**, uses

RL: NUU (Other use, unclassified); USES (Uses)

(method for reduction of residual organic solvent in carbomer)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 2 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 2003:666885 HCAPLUS
 DN 140:64873
 ED Entered STN: 27 Aug 2003
 TI Engineering micronization and **coating** applications with dense phase **carbon dioxide**
 AU Subramaniam, Bala
 CS Department of Chemical & Petroleum Engineering, The University of Kansas, Lawrence, KS, 66045-7609, USA
 SO Polymeric Materials Science and Engineering (2003), 89, 678
 CODEN: PMSEDG; ISSN: 0743-0515
 PB American Chemical Society
 DT Journal; (computer optical disk)
 LA English
 CC 63-5 (Pharmaceuticals)
 AB A process for producing and harvesting drug particles on a continuous basis using **supercrit. Carbon dioxide** (scCO₂) as an **antisolvent**, and a Wurster-type **coater** employing scCO₂ as the fluidizing medium and **antisolvent** are described. Particle micronization with scCO₂ allows for reproducible **crystal** formation with the potential for increased surface area and dissoln. rates. **Coating** with dense phase CO₂ allows the use of traditional organic soluble **coatings** with complete **solvent** recovery and virtually no atmospheric emissions. For particle micronization, ultrasonic energy is used to form droplets of drug solution. The scCO₂ selectively **exts.** the **solvent** from the droplets, **precipitating** the drug. The effluent from the **precipitation** chamber is led to a second high-pressure vessel where the particles are separated from the **solvent**-laden scCO₂. The micronization of several drugs including proteins and anti-cancer agents will be presented including anal. results such as the particle-size distribution, **crystallinity**, and residual **solvent** content. Advantages include the continuous production of virtually **solvent**-free drug particles in a narrow size range, CO₂ recycling, **solvent** recovery and ease of process scalability. For **coating** applications, glass inner and outer columns are housed in a high-pressure chamber in which dense phase CO₂ is used to fluidize the substrates. The CO₂ also removes the **solvent** from the **coating** solution sprayed on the substrates, thereby **precipitating** the **coating**. The system was used to **coat** a variety of substrates including tablets and stents for controlled release applications. This process **expands** the range of substrate/**coating** combinations possible with the Wurster **coater**, making it feasible to **coat** water-soluble substrates with **solutes** sprayed from organic solns.
 ST **supercrit carbon dioxide** fluidizing medium
antisolvent coating; dense phase **carbon dioxide** drug micronization
 IT **Solvents**
 (**antisolvents**; engineering micronization and **coating** applications with dense phase **carbon dioxide**)
 IT **Coating materials**
 (drug; engineering micronization and **coating** applications with dense phase **carbon dioxide**)
 IT Antitumor agents
 (engineering micronization and **coating** applications with dense phase **carbon dioxide**)
 IT Proteins
 RL: PEP (Physical, engineering or chemical process); PYP (Physical

process); PROC (Process)
 (engineering micronization and **coating** applications with
 dense phase **carbon dioxide**)

IT Pulverization
 (micronization; engineering micronization and **coating**
 applications with dense phase **carbon dioxide**)

IT Drugs
 (particles; engineering micronization and **coating**
 applications with dense phase **carbon dioxide**)

IT 124-38-9, **Carbon dioxide**, biological studies
 RL: BUU (Biological use, unclassified); BIOL (Biological study); USES
 (Uses)
 (**supercrit.**; engineering micronization and **coating**
 applications with dense phase **carbon dioxide**)

IT 124-38-9, **Carbon dioxide**, biological studies
 RL: BUU (Biological use, unclassified); BIOL (Biological study); USES
 (Uses)
 (**supercrit.**; engineering micronization and **coating**
 applications with dense phase **carbon dioxide**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 3 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:636787 HCAPLUS

ED Entered STN: 15 Aug 2003

TI Engineering micronization and **coating** applications with dense
 phase **carbon dioxide**

AU Subramaniam, Bala

CS Department of Chemical & Petroleum Engineering, The University of Kansas,
 Lawrence, KS, 66045-7609, USA

SO Abstracts of Papers, 226th ACS National Meeting, New York, NY, United
 States, September 7-11, 2003 (2003), PMSE-405 Publisher: American Chemical
 Society, Washington, D. C.
 CODEN: 69EKY9

DT Conference; Meeting Abstract

LA English

AB A process for producing and harvesting drug particles on a continuous
 basis using **supercrit. carbon dioxide**
 (scCO₂) as an **antisolvent**, and a Wurster-type **coater**
 employing scCO₂ as the fluidizing medium and **antisolvent** are
 described. Particle micronization with scCO₂ allows for reproducible
crystal formation with the potential for increased surface area
 and dissoln. rates. **Coating** with dense phase CO₂
 allows the use of traditional organic soluble **coatings** with complete
solvent recovery and virtually no atmospheric emissions. For particle
 micronization, ultrasonic energy is used to form droplets of drug solution
 The scCO₂ selectively **exts.** the **solvent** from the
 droplets, **precipitating** the drug. The effluent from the **precipitation**
 chamber is led to a second high-pressure vessel where the particles are
 separated from the **solvent**-laden scCO₂. The micronization of
 several drugs including proteins and anti-cancer agents will be presented
 including anal. results such as the particle-size distribution,
crystallinity, and residual **solvent** content. Advantages
 include the continuous production of virtually **solvent**-free drug
 particles in a narrow size range, CO₂ recycling, **solvent**
 recovery and ease of process scalability. For **coating**
 applications, glass inner and outer columns are housed in a high-pressure
 chamber in which dense phase CO₂ is used to fluidize the

substrates. The CO₂ also removes the **solvent** from the **coating** solution sprayed on the substrates, thereby **precipitating** the **coating**. The system was used to **coat** a variety of substrates including tablets and stents for controlled release applications. This process **expands** the range of substrate/**coating** combinations possible with the Wurster **coater**, making it feasible to **coat** water-soluble substrates with **solutes** sprayed from organic solns.

L98 ANSWER 4 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:424564 HCAPLUS

DN 138:390844

ED Entered STN: 04 Jun 2003

TI Preparation of superfine particles using fast **expanding supercritical** solution

IN Yin, Enhua

PA Huayu New-Type Electronics Material Co., Ltd., Peop. Rep. China

SO Faming Zhuanli Shenqing Gongkai Shuomingshu, 7 pp.

CODEN: CNXXEV

DT Patent

LA Chinese

IC ICM B01D009-02

ICS B01D011-00

CC 63-1 (Pharmaceuticals)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	CN 1344578	A	20020417	CN 2000-124564	20000922
PRAI	CN 2000-124564		20000922		
AB	The method comprises dissolving and swelling solute (such as aspirin, polylactic acid, stimulants, antiphlogistic, contraceptive, release-controlling agent, other polymer, or pigment) in CO ₂ (or metallic oxide in water) under supercrit. condition, ejecting, filtering , and settling. The equipment consists of solvent tank, high-pressure pump, heat exchanger, extraction reactor, pressure reactor, elec. control unit, nozzle, filter , flow gauge, and refrigerating machine.				
ST	fast expanding superfine particle supercrit soln				
IT	Supercritical fluids (fast expanding ; preparation of superfine particles using fast expanding supercrit. solution)				
IT	Drug delivery systems (particles, superfine; preparation of superfine particles using fast expanding supercrit. solution)				
IT	Anti-inflammatory agents Contraceptives Nervous system stimulants (preparation of superfine particles using fast expanding supercrit. solution)				
IT	124-38-9, Carbon dioxide , processes RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process) (preparation of superfine particles using fast expanding supercrit. solution)				
IT	50-78-2, Aspirin 26023-30-3, Poly[oxy(1-methyl-2-oxo-1,2-ethanediyl)] 26100-51-6, Polylactic acid RL: PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses) (preparation of superfine particles using fast expanding supercrit. solution)				
IT	124-38-9, Carbon dioxide , processes RL: PEP (Physical, engineering or chemical process); PYP (Physical				

process); PROC (Process)
 (preparation of superfine particles using fast **expanding**
supercrit. solution)

RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 5 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 2003:421466 HCAPLUS
 DN 140:133534
 ED Entered STN: 03 Jun 2003
 TI Application of dense **gas** techniques for the production of fine
 particles
 AU Foster, Neil R.; Dehghani, Fariba; Charoenchaitrakool, Kiang M.; Warwick,
 Barry
 CS School of Chemical and Industrial Chemistry, University of New South
 Wales, NSW 2052, Australia
 SO PharmSci (2003), 5(2), No pp. given
 CODEN: PHARFY; ISSN: 1522-1059
 URL: [http://www.pharmsci.org/view.asp?path=ps0502/ps050211/ps050211.xml&pd](http://www.pharmsci.org/view.asp?path=ps0502/ps050211/ps050211.xml&pd f=yes)
 f=yes
 PB American Association of Pharmaceutical Scientists
 DT Journal; (online computer file)
 LA English
 CC 63-5 (Pharmaceuticals)
 AB The feasibility of using dense **gas** techniques such as rapid
expansion of **supercrit.** solns. (RESS) and aerosol
solvent extraction system (ASES) for micronization of
 pharmaceutical compds. is demonstrated. The chiral nonsteroidal
 anti-inflammatory racemic ibuprofen is soluble in **carbon**
dioxide at 35°C and pressures above 90 bar. The particle
 size decreased to less than 2 µm while the degree of
crystallinity was slightly decreased when processed by RESS. The
 dissoln. rate of the ibuprofen (a poorly water-soluble compound) was
 significantly enhanced after processing by RESS. The nonsteroidal
 anti-inflammatory drug Cu₂(indomethacin)₄L₂(Cu-Indo); (L = DMF [DMF]),
 which possessed very low solubility in **supercrit.** CO₂, was
 successfully micronized by ASES at 25°C and 68.9 bar using DMF as
 the **solvent** and CO₂ as the **antisolvent**. The
 concentration of **solute** dramatically influenced the **precipitate**
 characteristics. The particles obtained from the ASES process were
 changed from bipyramidal to spherical, with particle size less than 5
 µm, as the concentration increased from 5 to 100 mg/g. A further increase in
solute concentration to 200 mg/g resulted in large porous spheres,
 between 20 and 50 µ, when processing Cu-Indo by the ASES method. The
 dissoln. rate of the micronized Cu-Indo was significantly higher than the
 com. product.
 ST ibuprofen micronization aerosol **solvent extn**
supercrit fluid dissoln **recrystn**; copper
 indomethacin micronization aerosol **solvent extn**
supercrit fluid dissoln; ophthalmic suspension copper
 indomethacin dissoln dense **gas** particle size
 IT **Solvent extraction**
 (aerosol; application of dense **gas** techniques for the production
 of fine particles)
 IT **Crystallinity**
 Dissolution
 Particle shape
 Particle size

Particle size distribution

Recrystallization

Solubility

Supercritical fluids

(application of dense **gas** techniques for the production of fine particles)

IT Pulverization

(micronization; application of dense **gas** techniques for the production of fine particles)

IT Drug delivery systems

(suspensions, ophthalmic; application of dense **gas** techniques for the production of fine particles)

IT 124-38-9, **Carbon dioxide**, uses 151-21-3, Sodium lauryl sulfate, uses

RL: NUU (Other use, unclassified); USES (Uses)

(application of dense **gas** techniques for the production of fine particles)

IT 15687-27-1, Ibuprofen 221357-17-1

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)

(application of dense **gas** techniques for the production of fine particles)

RE.CNT 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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IT 124-38-9, **Carbon dioxide**, uses

RL: NUU (Other use, unclassified); USES (Uses)

(application of dense **gas** techniques for the production of fine particles)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 6 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:287335 HCAPLUS

DN 138:293388

ED Entered STN: 15 Apr 2003

TI Spectroscopically Probing Microscopic **Solvent** Properties of Room-Temperature Ionic Liquids with the Addition of **Carbon Dioxide**

AU Lu, Jie; Liotta, Charles L.; Eckert, Charles A.

CS Schools of Chemical Engineering and Chemistry and Biochemistry and Specialty Separations Center, Georgia Institute of Technology, Atlanta, GA, 30332-0100, USA

SO Journal of Physical Chemistry A (2003), 107(19), 3995-4000

CODEN: JPCAFH; ISSN: 1089-5639

PB American Chemical Society

DT Journal

LA English

- CC 68-1 (Phase Equilibriums, Chemical Equilibriums, and Solutions)
Section cross-reference(s): 73, 76
- AB Room-temperature ionic liqs. (RTILs) provide an alternative for elimination of **solvent** emissions to the atmospheric for many reactions, but the subsequent separation of the products by conventional methods can be a challenge. However, the use of **supercrit. carbon dioxide** (scCO₂) as an **extractant** offers potential for a novel class of environmentally benign media for chemical reaction and downstream separation. The authors studied the **solvent** properties of mixts. of 1-butyl-3-Me imidazolium hexafluorophosphate ([bmim][PF₆]) and CO₂ as functions of temperature (35-50 °C) and CO₂ pressure (0-230 bar). They report the Kamlet-Taft dipolarity/polarizability parameter, volume **expansion**, and microviscosity. The results are consistent with a picture of local enhancement of RTIL composition around a chromophore, maintaining **solvent** strength even at fairly high loadings of CO₂, whereas the microviscosity in the vicinity of the **solute** is dramatically reduced, leading to enhanced mass transport and facilitated separation
- ST alkyl imidazolium fluorophosphate **carbon dioxide** mixt
solvent property solvatochromism
- IT Dielectric constant
Fluorescence
Green chemistry
Ionic liquids
Mass transfer
Microviscosity
Polarizability
Separation
Solvatochromism
(**solvent** properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with **carbon dioxide** as studied by solvatochromic and fluorescence probes)
- IT **Expansion**
(volume; **solvent** properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with **carbon dioxide** as studied by solvatochromic and fluorescence probes)
- IT 100-23-2, N,N-Dimethyl-4-nitroaniline 58293-56-4, DCVJ
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(**solvent** properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with **carbon dioxide** as studied by solvatochromic and fluorescence probes)
- IT 124-38-9, **Carbon dioxide**, properties
174501-64-5, 1-Butyl-3-methyl imidazolium hexafluorophosphate
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(**solvent** properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with **carbon dioxide** as studied by solvatochromic and fluorescence probes)
- RE.CNT 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD
- RE
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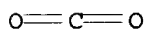
IT 124-38-9, Carbon dioxide, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)

(solvent properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with carbon dioxide as studied by solvatochromic and fluorescence probes)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L98. ANSWER 7 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:281854 HCAPLUS

DN 138:292772

ED Entered STN: 11 Apr 2003

TI Powder processing with pressurized gaseous fluids

IN Saim, Said; Horhota, Stephen; Koenig, Kenneth James; Bochniak, David Joseph

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO U.S. Pat. Appl. Publ., 37 pp.

CODEN: USXXCO

DT Patent

LA English

IC ICM B01D011-00

NCL 210634000; 210638000; 210644000; 210669000; 210702000; 210806000

CC 63-6 (Pharmaceuticals)

Section cross-reference(s): 48

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2003066800	A1	20030410	US 2002-268879	20021010
	WO 2003030871	A1	20030417	WO 2002-US32303	20021010

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,

UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,
 TJ, TM
 RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG,
 CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
 PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR,
 NE, SN, TD, TG

PRAI US 2001-328301P P 20011010

AB Disclosed is a method of small particle precipitation, retention and dispersion of

a solid or semi-solid material onto or into a carrier material. In the method, **solute** particles are precipitated from a pressurized gaseous fluid solution or a liquid solution and effectively retained and dispersed within

a carrier material. The technique can be advantageously used in pharmaceutical processing to produce a blend of solid or semi-solid material particles and carrier material, a granulation of the solid or semi-solid material particles with carrier material partially or fully coated with the solid and/or semi-solid material particles.

ST pharmaceutical particle pptn retention dispersion solid semisolid; pressurized gaseous fluid particle formation

IT Drug delivery systems

(capsules; method of particle precipitation and retention in carrier for processing)

IT Supercritical fluids

(for precipitation of **solute** in processing pharmaceutical particles)

IT Precipitation (chemical)

(powder processing with pressurized gaseous fluids)

IT Drug delivery systems

(tablets; method of particle precipitation and retention in carrier for processing)

IT 22254-24-6, Ipratropium bromide 30286-75-0, Oxytropium bromide

136310-93-5, Tiotropium bromide 174484-41-4, Tipranavir

RL: DMA (Drug mechanism of action); BIOL (Biological study)

(active material in processing pharmaceutical particles)

IT 9003-70-7, Polystyrene divinyl benzene 64044-51-5, Lactose monohydrate

RL: DMA (Drug mechanism of action); BIOL (Biological study)

(carrier for processing pharmaceutical particles)

IT 74-84-0, Ethane, uses 74-85-1, Ethylene, uses 74-98-6, Propane, uses

75-28-5, Isobutane 75-46-7, Trifluoromethane 106-97-8, Butane, uses

109-66-0, Pentane, uses 115-07-1, Propylene, uses 124-38-9,

Carbon dioxide, uses 2551-62-4, Sulfur hexafluoride

10024-97-2, Nitrous oxide, uses

RL: TEM (Technical or engineered material use); USES (Uses)

(for **solute** precipitation in producing of pharmaceutical particles)

IT 124-38-9, **Carbon dioxide**, uses

RL: TEM (Technical or engineered material use); USES (Uses)

(for **solute** precipitation in producing of pharmaceutical particles)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 8 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:227421 HCAPLUS

DN 138:227493

ED Entered STN: 25 Mar 2003

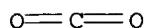
TI Solubilities of Imipramine HCl in **Supercritical Carbon Dioxide**

AU Jara-Morante, Eliana; Suleiman, David; Estevez, L. Antonio

CS Department of Chemical Engineering, University of Puerto Rico, Mayaguez,

00681-9046, P. R.
SO Industrial & Engineering Chemistry Research (2003), 42(8), 1821-1823
CODEN: IECRED; ISSN: 0888-5885
PB American Chemical Society
DT Journal
LA English
CC 68-1 (Phase Equilibriums, Chemical Equilibriums, and Solutions)
Section cross-reference(s): 45, 47, 63
AB The solubility of imipramine hydrochloride (I) in **supercrit. carbon dioxide** has been measured exptl. by a gravimetric technique. An ISCO **extraction** apparatus was modified to carry out the measurements. It consists of a syringe pump, a thermostatic chamber, an equilibrium cell, a variable-flow-rate restrictor, and an ice trap. Expts. were conducted by allowing the **supercrit. carbon dioxide** to slowly flow through the cell, where I had been previously loaded. The pressure was kept constant, controlled by the pump, and so was the flow rate, controlled by the restrictor. The amount of **solute** collected in the trap was measured in two different ways for consistency: gravimetrically and by dissolving the **solute** collected in methanol and measuring the concentration spectrophotometrically. The amount of **solvent** was measured by the difference in volume readings in the syringe pump (calculating the d. of **carbon dioxide** at the pump conditions); this value was also determined by measuring an average flow rate of the **expanded solvent** and the time of the run. A total of 52 measurements were done. Two five-point isotherms, at 40 and 50 °C, were obtained for pressures ranging from 30 to 50 MPa. Measured solubilities were within the range (5-10) + 10⁻⁶ mole fraction. These are the only published data for this system.
ST imipramine hydrochloride soly **supercrit carbon dioxide**
IT **Extraction apparatus**
(**extraction** set for determination of imipramine hydrochloride solubility in **supercrit. carbon dioxide**)
IT Solubility
(imipramine hydrochloride solubility in **supercrit. carbon dioxide**)
IT **Solvents**
(**supercrit.**; imipramine hydrochloride solubility in **supercrit. carbon dioxide**)
IT 113-52-0, Imipramine hydrochloride 124-38-9, **Carbon dioxide**, properties
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(imipramine hydrochloride solubility in **supercrit. carbon dioxide**)
RE.CNT 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
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(2) Larson, K; Biotechnol Prog 1986, V2(2), P73 HCAPLUS
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IT 124-38-9, **Carbon dioxide**, properties
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(imipramine hydrochloride solubility in **supercrit. carbon dioxide**)
RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L98 ANSWER 9 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2002:951622 HCAPLUS
DN 139:154684
ED Entered STN: 17 Dec 2002
TI **Crystal doping aided by rapid expansion of supercritical solutions**
AU Vemavarapu, Chandra; Mollan, Matthew J.; Needham, Thomas E.
CS Pharmaceutical Sci., pfizer Global R&D, Ann Arbor, MI, 48105, USA
SO AAPS PharmSciTech (2002), 3(4), No pp. given
CODEN: AAPHFZ; ISSN: 1522-1059
URL: <http://www.aapspharmscitech.org/scientificjournals/pharmscitech/volume3issue4/pt030429/pt030429.pdf>
PB American Association of Pharmaceutical Scientists
DT Journal; (online computer file)
LA English
CC 63-5 (Pharmaceuticals)
AB The purpose of this study was to test the utility of rapid **expansion of supercrit.** solution (RESS) based **cocrystns.** in inducing polymorph conversion and **crystal** disruption of chlorpropamide (CPD). CPD **crystals** were **recrystd.** by the RESS process utilizing **supercrit. carbon dioxide** as the **solvent**. The **supercrit.** region investigated for **solute extn** . ranged from 45 to 100°C and 2000 to 8000 psi. While pure **solute recrystn.** formed stage I of these studies, stage II involved **recrystn.** of CPD in the presence of urea (model impurity). The composition, morphol., and **crystallinity** of the particles thus produced were characterized utilizing techniques such as microscopy, thermal anal., x-ray powder diffractometry, and HPLC. Also, comparative evaluation between RESS and evaporative **crystallization** from liquid **solvents** was performed. RESS **recrystns.** of com. available CPD (form A) resulted in polymorph conversion to metastable forms C and V, depending on the temperature and pressure of the **recrystg . solvent**. **Cocrystn.** studies revealed the formation of eutectic mixts. and solid solns. of CPD + urea. Formation of the solid solns. resulted in the **crystal** disruption of CPD and subsequent amorphous conversion at urea levels higher than 40% wt/weight Consistent with these results were the redns. in m.p. (up to 9°C) and in the ΔH values of CPD (up to 50%). SEM revealed a particle size reduction of up to an order of magnitude upon RESS processing. Unlike RESS, **recrystns.** from liquid organic **solvents** lacked the ability to affect polymorphic conversions. Also, the incorporation of urea into the lattice of CPD was found to be inadequate. In providing the ability to control both the particle and **crystal** morphologies of active pharmaceutical ingredients, RESS proved potentially advantageous to **crystal** engineering. Rapid **crystallization** kinetics were found vital in making RESS-based doping superior to conventional **solvent** -based **cocrystns.**
ST chlorpropamide **crystal** doping **supercrit** soln
IT **Crystallization**
(**cocrystn.**; **crystal** doping aided by rapid **expansion of supercrit.** solns.)
IT Polymorphism (**crystal**)
Supercritical fluids
(**crystal** doping aided by rapid **expansion of supercrit.** solns.)

IT 57-13-6, Urea, uses
RL: MOA (Modifier or additive use); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses)
(crystal doping aided by rapid expansion of supercrit. solns.)

IT 94-20-2, Chlorpropamide
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(crystal doping aided by rapid expansion of supercrit. solns.)

IT 124-38-9, Carbon dioxide, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(crystal doping aided by rapid expansion of supercrit. solns.)

RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE

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(4) Burger, A; Sci Pharm 1975, V43, P152 HCAPLUS
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(29) Yu, L; J Pharm Sci 1995, V84(8), P966 HCAPLUS

IT 124-38-9, Carbon dioxide, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(crystal doping aided by rapid expansion of supercrit. solns.)

RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

ED Entered STN: 31 Jul 2002
TI Process for overcoming drug retention in hard gelatin inhalation capsules
AU Saim, Said; Horhota, Stephen T.
CS Boehringer Ingelheim Pharmaceuticals, Inc., Ridgefield, CT, 06877,
USA
SO Drug Development and Industrial Pharmacy (2002), 28(6), 641-654
CODEN: DDIPD8; ISSN: 0363-9045
PB Marcel Dekker, Inc.
DT Journal
LA English
CC 63-6 (Pharmaceuticals)
AB The quantity and consistency of drug delivery from dry powder inhalation devices that incorporate a pre-measured dose in a hard shell capsule of gelatin or other compatible material can be neg. affected by mold release lubricants used in capsule manufacturing This paper describes a novel process employing supercrit. CO2 for selective extraction of the fraction of lubricant responsible for the observed high and inconsistent drug retention in capsules and the ensuing lack of reproducibility of drug delivery. The process allows for lubricant removal from seemingly inaccessible interior surfaces of assembled capsule shells without altering the structural or chemical properties of the capsules. Diffusion limitations are overcome through repeated pressure increase and decrease to generate significant convective flow of dissolved lubricant out of the capsule. Drug retention is alleviated only if nearly all the retentive fraction of the lubricant is removed. The effect of extraction with supercrit. CO2 on the structure of the internal surfaces of the capsules is investigated using SEM. Key performance parameters such as drug and carrier retention and fine particle mass are investigated using simulated inhalation tests. Laboratory and pilot scale extns. yielded similar results.
ST drug retention inhaler gelatin capsule lubricant
IT Drug delivery systems
(capsules; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
IT Medical goods
(inhalers; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
IT Lubricants
(overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
IT Drug delivery systems
(powders, inhalants; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
IT Extraction
(supercrit.; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
IT 63-42-3, Lactose 22254-24-6, Ipratropium bromide
RL: PRP (Properties); THU (Therapeutic use); BIOL (Biological study); USES (Uses)
(overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
IT 124-38-9, Carbon dioxide, biological studies
RL: BSU (Biological study, unclassified); BIOL (Biological study)
(supercrit.; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
RE.CNT 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
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1993

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 IT 124-38-9, **Carbon dioxide**, biological studies
 RL: BSU (Biological study, unclassified); BIOL (Biological study)
 (supercrit.; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

- L98 ANSWER 11 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 2002:267861 HCAPLUS
 DN 137:125601
 ED Entered STN: 10 Apr 2002
 TI Effects of **solvent** density on **retention** in **gas**
 -liquid chromatography I. Alkanes **solutes** in polyethylene glycol stationary phases
 AU Gonzalez, F. R.; Perez-Parajon, J.; Garcia-Dominguez, J. A.
 CS Instituto de Quimica-Fisica Rocasolano, CSIC, Madrid, 28006, Spain
 SO Journal of Chromatography, A (2002), 953(1-2), 151-163
 CODEN: JCRAEY; ISSN: 0021-9673
 PB Elsevier Science B.V.
 DT Journal
 LA English
 CC 36-5 (Physical Properties of Synthetic High Polymers)
 Section cross-reference(s): 66, 80
 AB **Gas**-liquid chromatog. columns were prepared by **coating** silica capillaries with poly(oxyethylene) polymers of different mol. mass distributions, in the range of low number-average molar masses, where the d. still varies significantly. A novel, high-temperature, rapid evaporation method was developed and applied to the static **coating** of the low-mol.-mass stationary phases. The anal. of alkanes **retention** data from these columns reveals that the dependence of the partition coefficient with the **solvent** macroscopic d. is mainly due to a variation of entropy. Enthalpies of **solute** transfer contribute poorly to the observed variations of **retention**. Since the alkanes solubility diminishes with the increasing **solvent** d., and this variation is weakly dependent with temperature, it is concluded that the decrease of free-volume in the liquid is responsible for this behavior.
 ST polyethylene glycol **gas** liq chromatog density; alkane **retention** thermodyn property chromatog polyethylene glycol
 IT Polyoxyalkylenes, processes
 RL: PEP (Physical, engineering or chemical process); POF (Polymer in formulation); PYP (Physical process); TEM (Technical or engineered material use); PROC (Process); USES (Uses)
 (Carbowax 1000, Carbowax 1540; alkanes **solutes** in polyethylene glycol stationary phases **gas**-liquid chromatog.)
 IT **Gas** chromatography
 (alkanes **solutes** in polyethylene glycol stationary phases **gas**-liquid chromatog.)
 IT Alkanes, properties
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
 (alkanes **solutes** in polyethylene glycol stationary phases **gas**-liquid chromatog.)
 IT Thermal **expansion**

- (coefficient; of polyethylene glycol stationary phases on alkanes **retention** in gas-liquid chromatog.)
- IT Heat capacity
Partition
Transfer enthalpy
Transfer entropy
(of alkanes **solutes** in polyethylene glycol stationary phases **gas-liquid chromatog.**)
- IT Density
Molecular weight
(of polyethylene glycol stationary phases on alkanes **retention** in **gas-liquid chromatog.**)
- IT 25322-68-3, Carbowax 600
RL: PEP (Physical, engineering or chemical process); POF (Polymer in formulation); PYP (Physical process); TEM (Technical or engineered material use); PROC (Process); USES (Uses)
(Carbowax 1000, Carbowax 1540; alkanes **solutes** in polyethylene glycol stationary phases **gas-liquid chromatog.**)
- IT 208196-86-5, HP-Innowax 318235-13-1, AT-wax
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); TEM (Technical or engineered material use); PROC (Process); USES (Uses)
(alkanes **solutes** in crosslinked polyethylene glycol stationary phases **gas-liquid chromatog.**)
- IT 7631-86-9, Silica, miscellaneous
RL: MSC (Miscellaneous)
(alkanes **solutes** in polyethylene glycol stationary phases **gas-liquid chromatog.**)
- IT 111-84-2, n-Nonane 112-40-3, n-Dodecane 124-18-5, n-Decane 629-50-5, n-Tridecane 629-59-4, n-Tetradecane 1120-21-4, n-Undecane
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(alkanes **solutes** in polyethylene glycol stationary phases **gas-liquid chromatog.**)

RE.CNT 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD

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L98 ANSWER 12 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 2001:703010 HCAPLUS
 DN 135:247285
 ED Entered STN: 26 Sep 2001
 TI Method for extraction and reaction using supercritical fluids
 IN Horhota, Stephen T.; Saim, Said
 PA Boehringer Ingelheim Pharmaceuticals, Inc., USA
 SO U.S., 17 pp., Cont.-in-part of U.S. 6,228,394.
 CODEN: USXXAM
 DT Patent
 LA English
 IC ICM A61K009-64
 ICS A61K009-48; F26B003-00
 NCL 424456000
 CC 63-8 (Pharmaceuticals)
 FAN.CNT 3

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 6294194	B1	20010925	US 2000-517883	20000303
	US 6228394	B1	20010508	US 1998-157267	19980921
	ZA 9809261	A	19990531	ZA 1998-9261	19981012
	WO 2001066214	A1	20010913	WO 2001-US2356	20010125
	W: AU, BR, CA, CN, CZ, HU, IL, IN, JP, KR, MX, NZ, PL, RU, TR, ZA				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
	EP 1265683	A1	20021218	EP 2001-908680	20010125
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
	BR 2001008855	A	20030429	BR 2001-8855	20010125
	JP 2003525730	T2	20030902	JP 2001-564860	20010125
	US 2001036898	A1	20011101	US 2001-879031	20010612
	US 6610624	B2	20030826		
	ZA 2002006989	A	20030407	ZA 2002-6989	20020830
	US 2004014590	A1	20040122	US 2003-620398	20030716
PRAI	US 1997-62099P	P	19971014		
	US 1998-157267	A2	19980921		
	US 2000-517883	A	20000303		
	WO 2001-US2356	W	20010125		
	US 2001-879031	A3	20010612		

AB Methods for removing soluble material from confined spaces within substrates such as containers, capsules and porous powders comprising extraction with supercrit. fluids, the pressure of which is preferably modulated between an upper level and a lower level within a relatively narrow range of fluid pressure and d. The method permits enhanced extraction efficiency, catalytic reaction rates and ability to maintain catalyst activity. A small amount of polyethylene glycol with an average mol. weight of 200 was pipetted into a 1-mL capped vial and the cap was pierced with a 500 mm needle. The level of the polymer was about 1/4" above the bottom of the vial. The polymer was then extracted at either a constant pressure of 165 bar or using the pressure modulation technique in the range of 159-172 bar. Temperature and extraction

time

were 35° and 58 min resp. in both runs. Despite small pressure and d. modulation, the modulation technique was substantially more efficient at removing PEG 200 from the capped vial than conventional SFE. Extraction efficiency was nearly 7-fold higher than that of conventional SFE. The ability to rapidly modulate pressure appears to allow for very high extraction efficiency when compared to conventional SFE.

ST extn reaction supercrit fluid
 IT Drug delivery systems
 (capsules; method for extraction and reaction using supercrit. fluids)
 IT Density
 Extraction
 Lubricants
 Nutrients
 Plasticizers
 Pressure
 Supercritical fluids
 Temperature
 (method for extraction and reaction using supercrit. fluids)
 IT Polyoxyalkylenes, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (method for extraction and reaction using supercrit. fluids)
 IT 64-17-5, Ethanol, uses 124-38-9, carbon
 dioxide, uses 25322-68-3, polyethylene glycol
 RL: NUU (Other use, unclassified); USES (Uses)
 (method for extraction and reaction using supercrit. fluids)
 IT 9005-25-8, starch, biological studies 22254-24-6, ipratropium bromide
 RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses)
 (method for extraction and reaction using supercrit. fluids)

RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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 - (2) Anon; WO 9949996 1999
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- IT 124-38-9, carbon dioxide, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (method for extraction and reaction using supercrit. fluids)

RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 13 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:676650 HCAPLUS

DN 135:244446

ED Entered STN: 14 Sep 2001

TI Material processing by repeated solvent expansion-contraction

IN Saim, Said; Horhota, Stephen; Bochniak, David Joseph

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO PCT Int. Appl., 48 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM B01D011-02

ICS B01D009-00

CC 48-1 (Unit Operations and Processes)

Section cross-reference(s): 63

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001066215	A1	20010913	WO 2001-US3019	20010130
	W: AU, BR, CA, CN, CZ, HU, IL, IN, JP, KR, MX, NZ, PL, RU, TR, US, ZA				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
	US 2001055561	A1	20011227	US 2001-774232	20010130
	EP 1263516	A1	20021211	EP 2001-906792	20010130
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
	BR 2001008912	A	20021224	BR 2001-8912	20010130
	JP 2003525731	T2	20030902	JP 2001-564861	20010130
	ZA 2002006943	A	20030404	ZA 2002-6943	20020829
PRAI	US 2000-186888P	P	20000303		
	WO 2001-US3019	W	20010130		

AB A method is disclosed for repeatedly converting a solvent from a state of solvent to a state of antisolvent with relatively little loss of solvent. The method is used to allow for processing of large amts. of solute material with min. amts. of solvent.

ST solute material processing solvent expansion contraction

IT Solvents

(antisolvents; material processing by repeated solvent expansion-contraction)

IT Coating process

Contraction (mechanical)

Crystallization

Drugs

Expansion

Extraction

Recycling

Solutes

Solvents

(material processing by repeated solvent expansion-contraction)

IT 64-17-5, Ethanol, uses 67-68-5, DmsO, uses 103-90-2, Acetaminophen

124-38-9, Carbon dioxide, uses

RL: TEM (Technical or engineered material use); USES (Uses)
(material processing by repeated solvent expansion-contraction)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE

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- (3) Davis, T; US 4536283 A 1985 HCAPLUS
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- (5) Upjohn Co; WO 9003782 A 1990 HCAPLUS

IT **124-38-9, Carbon dioxide, uses**

RL: TEM (Technical or engineered material use); USES (Uses)
(material processing by repeated solvent expansion-contraction)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 14 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN **2001:676649** HCAPLUS

DN **135:231760**

ED Entered STN: 14 Sep 2001

TI Methods for extraction of drugs and related materials using supercritical fluids

IN Horhota, Stephen T.; Saim, Said

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO PCT Int. Appl., 54 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM B01D011-02

CC 63-8 (Pharmaceuticals)

Section cross-reference(s): 17, 48

FAN.CNT 3

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001066214	A1	20010913	WO 2001-US2356	20010125
W: AU, BR, CA, CN, CZ, HU, IL, IN, JP, KR, MX, NZ, PL, RU, TR, ZA				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
US 6294194	B1	20010925	US 2000-517883	20000303
EP 1265683	A1	20021218	EP 2001-908680	20010125
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
BR 2001008855	A	20030429	BR 2001-8855	20010125
JP 2003525730	T2	20030902	JP 2001-564860	20010125
PRAI US 2000-517883	A	20000303		
US 1997-62099P	P	19971014		
US 1998-157267	A2	19980921		
WO 2001-US2356	W	20010125		

AB Methods for removing soluble material from confined spaces within substrates such as containers, capsules and porous powders comprise extraction with supercrit. fluids, the pressure of which is preferably modulated between an upper level and a lower level within a relatively narrow range of fluid pressure and d. The method permits enhanced extraction efficiency, catalytic reaction rates and ability to maintain catalyst activity. The capsules containing a drug were extracted by using CO₂ at 65° at 552 bar.

ST supercrit fluid extn drug container

IT Drug delivery systems
(capsules; extraction of drugs and related materials using supercrit. fluids)

IT Absorbents
Adsorbents
Catalysts
Containers
Drugs
Extraction apparatus
Lubricants
Plasticizers
Vials
(extraction of drugs and related materials using supercrit. fluids)

IT Polyoxyalkylenes, processes
RL: PEP (Physical, engineering or chemical process); PROC (Process)
(extraction of drugs and related materials using supercrit. fluids)

IT Extraction
(supercrit.; extraction of drugs and related materials using supercrit. fluids)

IT 64-17-5, Ethanol, processes 25322-68-3, Polyethylene glycol
RL: PEP (Physical, engineering or chemical process); PROC (Process)
(extraction of drugs and related materials using supercrit. fluids)

IT 22254-24-6, Ipratropium bromide
RL: PEP (Physical, engineering or chemical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(extraction of drugs and related materials using supercrit. fluids)

IT 124-38-9, Carbon dioxide, uses
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
(supercrit.; extraction of drugs and related materials using supercrit. fluids)

RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Barton, J; WO 9949996 A 1999

(2) Boehringer Ingelheim Pharma; WO 9918939 A 1999 HCAPLUS

(3) Krukonis, V; US 5514220 A 1996

(4) Saim, S; US 5725756 A 1998 HCAPLUS

IT 124-38-9, Carbon dioxide, uses
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
(supercrit.; extraction of drugs and related materials using supercrit. fluids)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 15 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:664895 HCAPLUS

DN 135:262853

ED Entered STN: 12 Sep 2001

TI Predicting solubility in **supercritical solvents** using estimated virial coefficients and fluctuation theory

AU Tomberli, B.; Goldman, S.; Gray, C. G.

CS University of Guelph, Guelph-Waterloo Physics Institute, Guelph, ON, N1G 2W1, Can.

SO Fluid Phase Equilibria (2001), 187-188, 111-130
CODEN: FPEQDT; ISSN: 0378-3812

PB Elsevier Science B.V.

DT Journal

LA English

CC 68-1 (Phase Equilibriums, Chemical Equilibriums, and Solutions)
Section cross-reference(s): 65, 69

AB A theor. method based on combining the virial **expansion** and fluctuation theory for calculating the chemical potential of a **solute** in a **supercrit. fluid** is presented. The method is compared to literature results from Monte Carlo simulations based the Widom method for evaluating the chemical potential. For one-center and two-center Lennard Jones (2CLJ) potential models, the average difference from simulated results for the chemical potential is about 5 at densities up to twice the critical d. The method requires virial coeffs. up to C(T) (the third) to achieve this level of accuracy. Correlations based on corresponding states principles for the prediction of B(T) [AIChE J. 20 (1974) 263; AIChE J. 21 (1975) 827; AIChE J. 24 (1978) 1978] and C(T) [AIChE J. 29 (1983) 107] are used to estimate these virial coeffs. A comparison with exptl. determined values for naphthalene in **carbon dioxide** shows the ests. to be accurate at typical **supercrit. extraction** conditions. These correlations are then used to determine virial coeffs. and chemical potentials for naphthalene, benzoic acid and phenanthrene in **carbon dioxide** at several different state conditions for which solubility data exist. The theor. results are compared to chemical potentials obtained from exptl. solubility data.

The method is found to be accurate, tractable and systematically improvable through the inclusion of higher order terms in the virial **expansion**.

ST **solute supercrit solvent** soly virial coeff
fluctuation theory

IT Statistical mechanics
(fluctuation theory; **solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)

IT Chemical potential
Critical density
Lennard-Jones potential
Pair potential
Solubility
Solutes
Virial coefficient
(**solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)

IT **Extraction**
Solvents
(**supercrit.**; **solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)

IT 65-85-0, Benzoic acid, properties 85-01-8, Phenanthrene, properties 91-20-3, Naphthalene, properties 124-38-9, **Carbon dioxide**, properties
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
(**solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)

RE.CNT 45 THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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- (2) Anon; Design Institute for Physical Property Data American Institute of Chemical Engineers, Physical and Thermodynamic Properties of Pure Chemicals 1989
- (3) Barker, J; J Chem Phys 1966, V44, P4206 HCAPLUS
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- (5) Bohm, H; Mol Phys 1984, V53, P651
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- IT 124-38-9, Carbon dioxide, properties
 RL: PEP (Physical, engineering or chemical process); PRP (Properties);
 PROC (Process)
 (solute solubility in supercrit. solvents from
 estimated virial coeffs. and fluctuation theory)
- RN 124-38-9 HCAPLUS
- CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 16 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 2001:107664 HCAPLUS
 DN 134:285514
 ED Entered STN: 13 Feb 2001
 TI Green process concepts for the pharmaceutical industry
 AU Subramaniam, Bala; Saim, Said; Rajewski, Roger; Stella,
 Valentino J.
 CS Department of Chemical and Petroleum Engineering, University of Kansas,
 Lawrence, KS, 66045-2223, USA

- SO ACS Symposium Series (2001), 766 (Green Engineering), 96-110
CODEN: ACSMC8; ISSN: 0097-6156
- PB American Chemical Society
- DT Journal; General Review
- LA English
- CC 63-0 (Pharmaceuticals)
- AB A review with 23 refs. Process concepts for producing drug particles using **supercrit. carbon dioxide** (scCO₂) as an **antisolvent** and for substrate **coating** employing scCO₂ as the fluidizing medium and **antisolvent** are described. Particle micronization with scCO₂ allows for reproducible **crystal** formation with the potential for increased surface area and dissoln. rates. **Coating** with scCO₂ allows the use of traditional organic-soluble **coatings** with complete **solvent** recovery and virtually no atmospheric emissions. For formation of drug nanoparticles, an ultrasonic nozzle that employs scCO₂ as the energizing medium is used to form droplets of the drug-laden solution. The scCO₂ also selectively **exs.** the **solvent** from the droplets, **precipitating** the drug. Submicron particles of hydrocortisone and ibuprofen (600 nm or less) formed in this manner are presented. Advantages include the production of virtually **solvent**-free drug particles in a narrow size range. For particle **coating**, scCO₂ is used to fluidize the core substrate particles. The scCO₂ also removes the **solvent** from the **coating** solution sprayed on the substrates, thereby **precipitating** the **coating**. This **coating** process **expands** the range of substrate/**coating** combinations possible with the conventional air-suspension Wurster **coater**, making it feasible to **coat** water-soluble substrates with **solutes** sprayed from organic solns.
- ST review pharmaceutical industry green process
- IT Pharmaceutical industry
(green process concepts for pharmaceutical industry)
- IT 124-38-9, **Carbon dioxide**, uses
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
(**supercrit.**; green process concepts for pharmaceutical industry)
- RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD
- RE
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 - (7) Gallagher, P; ACS Symposium Series 1989, 406, P334
 - (8) Hartmann, J; Journal of Scientific Instruments 1939, V16, P140
 - (9) Kreuter, J; J Controlled Release 1989, V16, P169
 - (10) Matson, D; Ind Eng Chem Res 1987, V26, P2298 HCAPLUS
 - (11) Mawson, S; J Appl Polym Sci 1997, V64, P2105 HCAPLUS
 - (12) Muller, B; German Patent Appl No DE 3744329 A1 1989
 - (13) Randolph, T; Biotechnol Prog 1993, V9, P429 HCAPLUS
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 - (17) Subramaniam, B; US 5833891 1998 HCAPLUS
 - (18) Subramaniam, B; J Pharm Sci 1997, V86, P885 HCAPLUS
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(22) Yeo, S; Biotech Bioeng 1993, V41, P341 HCAPLUS
 (23) York, P; Respiratory Drug Delivery V: Program and Proceedings 1996, P231 HCAPLUS
 IT 124-38-9, Carbon dioxide, uses
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
 (supercrit.; green process concepts for pharmaceutical industry)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 17 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1999:806362 HCAPLUS
 DN 132:109880
 ED Entered STN: 22 Dec 1999
 TI **Supercritical fluid extraction** of solids.
 Statistical thermodynamic approach
 AU Boublik, Tomas
 CS Department of Physical and Macromolecular Chemistry, Charles University, Faculty of Science, Prague, Czech Rep.
 SO Physical Chemistry Chemical Physics (2000), 2(1), 91-95
 CODEN: PPCPFQ; ISSN: 1463-9076
 PB Royal Society of Chemistry
 DT Journal
 LA English
 CC 48-1 (Unit Operations and Processes)
 Section cross-reference(s): 69
 AB To study the effect of non-sphericity of **solvent** and **solute** mols. on the main characteristics of **supercrit. fluid extraction** the fourth-order virial **expansion** is considered in which the individual virial coeffs. (and cross terms) are determined from the formula proposed recently for the Kihara generalized pair potential. The Kihara four-step square-well potential is assumed; its form makes it possible to write analytic expressions for the considered virial coeffs. and, consequently, for the main thermodyn. functions - the residual chemical potential of **solute** and total pressure. The method is applied to determine the dependence of the mole fraction of **solute** on temperature or pressure in the binary systems **carbon dioxide-naphthalene** and **ethylene-naphthalene** and the effect of the **cosolvent** on the **solute** concentration in the system **ethylene-naphthalene-acetone** at 308 K. Fair agreement with the simulation and exptl. data was found.
 ST **supercrit fluid extn** solid statistical thermodyn analysis; **carbon dioxide** naphthalene **supercrit fluid extn**; **ethylene** naphthalene **supercrit fluid extn**
 IT Solids
 Statistical thermodynamics
 (statistical thermodyn. approach in **supercrit. fluid extraction** of solids)
 IT **Extraction**
 (**supercrit.**; statistical thermodyn. approach in **supercrit. fluid extraction** of solids)
 IT 124-38-9, Carbon dioxide, processes
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); TEM (Technical or engineered material use); PROC (Process); USES (Uses)
 (statistical thermodyn. approach in **supercrit. fluid extraction** of solids)

IT 67-64-1P, Acetone, processes 74-85-1P, Ethylene, processes 91-20-3P, Naphthalene, processes

RL: PUR (Purification or recovery); REM (Removal or disposal); PREP (Preparation); PROC (Process)

(statistical thermodyn. approach in **supercrit. fluid extraction** of solids)

RE.CNT 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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- (11) McHugh, M; Supercritical Fluid Extraction 1994
- (12) Pavlicek, J; J Phys Chem 1992, V98, P2298
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- (16) Tsekhanskaya, Y; Zh Fiz Khim 1964, V38, P2106

IT 124-38-9, Carbon dioxide, processes

RL: PEP (Physical, engineering or chemical process); PRP (Properties); TEM (Technical or engineered material use); PROC (Process); USES (Uses)

(statistical thermodyn. approach in **supercrit. fluid extraction** of solids)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 18 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1999:262169 HCAPLUS

DN 130:301697

ED Entered STN: 29 Apr 1999

TI Methods of treating capsules and dry, powdered pharmaceutical formulations

IN Horhota, Steven T.; Said, Saim

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO PCT Int. Appl., 79 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM A61K009-48

ICS A61K009-14

CC 63-6 (Pharmaceuticals)

FAN.CNT 3

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9918939	A1	19990422	WO 1998-US20815	19981005
	W: AU, BG, BR, CA, CN, CZ, EE, HU, IL, JP, KR, LT, LV, MX, NZ, PL, RO, RU, SI, TR				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	US 6228394	B1	20010508	US 1998-157267	19980921
	CA 2302276	AA	19990422	CA 1998-2302276	19981005
	AU 9897838	A1	19990503	AU 1998-97838	19981005
	AU 753076	B2	20021010		

EP 1024794 A1 20000809 EP 1998-952043 19981005
 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
 IE, SI, LT, LV, FI, RO

BR 9814818 A 20001003 BR 1998-14818 19981005
 EE 200000292 A 20010815 EE 2000-200000292 19981005
 JP 2001519380 T2 20011023 JP 2000-515575 19981005
 NZ 504394 A 20021220 NZ 1998-504394 19981005
 ZA 9809261 A 19990531 ZA 1998-9261 19981012
 MX 200003329 A 20001110 MX 2000-3329 20000405
 BG 104317 A 20001229 BG 2000-104317 20000407

PRAI US 1997-62099P P 19971014
 US 1998-157267 A 19980921
 WO 1998-US20815 W 19981005

AB Undesirable materials present in gelatin, cellulose or plastic capsules
 used for storing a dry, powdered pharmaceutical formulation are extracted by
 supercrit. fluids. The method is also used for removing undesirable
 material from drug powder. The amount of powder retained in the capsules
 following inhalation is minimized. A powder blend of lactose and
 ipratropium bromide was loaded into CO₂-treated capsules and
 significant reduction in the amts. of drug or carrier retained in the capsules
 following inhalation was demonstrated.

ST supercrit fluid extn capsule impurity removal; ipratropium bromide lactose
 powder inhalant

IT Drug delivery systems
 (capsules; removal of impurities from capsules containing powdery
 ingredients by supercrit. fluid extraction)

IT Drug delivery systems
 (inhalants; removal of impurities from capsules containing powdery
 ingredients by supercrit. fluid extraction)

IT Lubricants
 Water vapor
 (removal of impurities from capsules containing powdery ingredients by
 supercrit. fluid extraction)

IT Extraction
 (supercrit.; removal of impurities from capsules containing powdery
 ingredients by supercrit. fluid extraction)

IT 124-38-9, Carbon dioxide, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (removal of impurities from capsules containing powdery ingredients by
 supercrit. fluid extraction)

IT 63-42-3, Lactose 22254-24-6, Ipratropium bromide
 RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses)
 (removal of impurities from capsules containing powdery ingredients by
 supercrit. fluid extraction)

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Genentech Inc; US 5641510 A HCAPLUS
 (2) Genentech Inc; WO 9601105 A 1996 HCAPLUS
 (3) Heit; US 5287632 A 1994
 (4) Minnesota Mining And Manufacturing Company; WO 9518834 A 1995 HCAPLUS
 (5) Sumitomo Heavy Industries; DE 3545913 A 1986 HCAPLUS
 (6) Syntex U S A Inc; EP 0421577 A 1991 HCAPLUS

IT 124-38-9, Carbon dioxide, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (removal of impurities from capsules containing powdery ingredients by
 supercrit. fluid extraction)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

L98 ANSWER 19 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1998:731765 HCAPLUS
 DN 129:347344
 ED Entered STN: 18 Nov 1998
 TI Methods for a particle precipitation and coating using near-critical and supercritical antisolvents
 IN Subramaniam, Bala; Saim, Said; Rajewski, Roger A.; Stella, Valentino
 PA The University of Kansas, USA
 SO U.S., 30 pp., Cont.-in-part of U.S. Ser. No. 722,463.
 CODEN: USXXAM
 DT Patent
 LA English
 IC ICM B01B011-00
 ICS B01J002-04; B05D001-02
 NCL 264007000
 CC 63-8 (Pharmaceuticals)
 FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 5833891	A	19981110	US 1997-805215	19970227
	US 5874029	A	19990223	US 1996-723463	19961009
	CA 2247900	AA	19970904	CA 1997-2247900	19970228
	WO 9731691	A1	19970904	WO 1997-US3207	19970228
	W:		AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM		
	RW:		GH, KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG		
	AU 9721936	A1	19970916	AU 1997-21936	19970228
	EP 709384	B2	19990826		
	EP 885038	A2	19981223	EP 1997-914827	19970228
	R:		AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI		
	JP 2002504011	T2	20020205	JP 1997-531174	19970228
PRAI	US 1996-723463	A2	19961009		
	US 1996-12592P	P	19960301		
	US 1996-12593P	P	19960301		
	US 1997-805215	A	19970227		
	WO 1997-US3207	W	19970228		

AB Improved methods and apparatus for particle precipitation and coating using near- or

supercrit. fluid conditions are described. A fluid dispersion having a continuous phase dispersant and at least one precipitable substance therein is contacted with a supercrit. fluid (SCF) antisolvent so as to generate focused high frequency antisolvent sonic waves, breaking up the dispersion into extremely small droplets; the enhanced mass transfer rates between the droplets and the antisolvent causes precipitation of very small particles on the order of 0.1-10 μ m. In coating processes, a turbulent fluidized flow of core particles is created using an SCF antisolvent in an enclosed zone. The core particles are contacted therein at near- or supercrit. conditions by a fluid dispersion containing a dispersant together with a precipitable substance. The antisolvent depletes the dispersant and the substance is precipitated onto the fluidized core particles. In

another

aspect of the invention, a process for preparing and administering a medicament using only a single container is provided. In such method, a fluid dispersion having a dispersant with the medicament therein is contacted with an antisolvent at near- or supercrit. conditions within a

in use container, so as to directly precipitate small particles of the medicament

the container. The antisolvent is then removed and the use container is sealed with the medicament particles therein. Thereafter, dose(s) of the medicament can be withdrawn from the use container and administered to a patient. Examples are given for recrystn. of hydrocortisone, RG503H, ibuprofen, or camptothecin from a DMSO solution using compressed CO₂ as energizing gas and antisolvent.

ST recrystn drug particle pptn supercrit antisolvent

IT **Solvents**

(**antisolvents**; particle precipitation and coating using near-critical and **supercrit. antisolvents**)

IT Electromagnetic wave

(high-frequency; particle precipitation and coating using near-critical and supercrit. antisolvents)

IT Coating materials

Disperse systems

Dispersing agents

Particle size

Particles

Recrystallization

Supercritical fluids

(particle precipitation and coating using near-critical and supercrit. antisolvents)

IT 74-98-6, Propane, properties 75-28-5, Isobutane 75-46-7,

Trifluoromethane 106-97-8, Butane, properties 124-38-9,

Carbon dioxide, properties 2551-62-4, Sulfur

hexafluoride 10024-97-2, Nitrous oxide, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties);

PROC (Process)

(antisolvent and energizing gas; particle precipitation and coating using near-critical and supercrit. antisolvents)

IT 7440-59-7, Helium, properties 7727-37-9, Nitrogen, properties

7782-44-7, Oxygen, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties);

PROC (Process)

(energizing gas; particle precipitation and coating using near-critical and supercrit. antisolvents)

IT 50-23-7, Hydrocortisone 67-68-5, DmsO, properties 7689-03-4,

Camptothecin 15687-27-1, Ibuprofen 34346-01-5, Glycolic acid-lactic acid copolymer

RL: PEP (Physical, engineering or chemical process); PRP (Properties);

PROC (Process)

(particle precipitation and coating using near-critical and supercrit. antisolvents)

RE.CNT 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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(2) Anon; EP 0542314 1993 HCAPLUS

(3) Anon; WO 9501221 1995

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(6) Barry; US 4900558 1990 HCAPLUS

(7) Bodmeier; Pharmaceutical Research 1995, V12(8) HCAPLUS

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(11) Dixon; Polymer 1994, V35(18) HCAPLUS

(12) Fischer; US 5043280 1991 HCAPLUS

(13) Gallagher; US 5389263 1995 HCAPLUS

(14) Kim; US 5344676 1994

(15) Kurkonis; US 5360478 1994 HCAPLUS

- (16) Lefebvre; Atomization and Sprays 1989, P136
(17) Niwa; Journal of Controlled Release 1993, V25, P89 HCAPLUS
(18) Prince; US 5308648 1994 HCAPLUS
(19) Randolph; Biotechnol Prog 1993, V9(4) HCAPLUS
(20) Sanchez; International Journal of Pharmaceutics 1993, V99, P263 HCAPLUS
(21) Sievers; US 5301664 1994
(22) Tom; Biotechnol 1991, V7, P403 HCAPLUS
(23) Wilcox; A I Ch E Journal 1965, V11(1), P69 HCAPLUS
(24) Yeo; Biotechnology and Bioengineering 1993, V41, P341 HCAPLUS
(25) Yeo; J Pharmaceutical Sciences 1994, V83(12) HCAPLUS
(26) Yeo; Macromolecules 1993, V26, P6207 HCAPLUS
(27) York; Respiratory Drug Delivery V 1996, P231 HCAPLUS
IT 124-38-9, Carbon dioxide, properties
RL: PEP (Physical, engineering or chemical process); PRP (Properties);
PROC (Process)
(antisolvent and energizing gas; particle precipitation and coating using
near-critical and supercrit. antisolvents)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

- L98 ANSWER 20 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1998:140144 HCAPLUS
ED Entered STN: 09 Mar 1998
TI The behavior of micellar systems formed in **supercritical**
carbon dioxide and their use as nanobioreactors.
AU Niemeyer, E. D.; Bonzagni, N. J.; Bright, F. V.
CS Department Chemistry, State University New York, Buffalo, NY, 14260-3000,
USA
→ SO Book of Abstracts, 215th ACS National Meeting, Dallas, March 29-April 2
(1998), PHYS-251 Publisher: American Chemical Society, Washington, D. C.
CODEN: 65QTAA
DT Conference; Meeting Abstract
LA English
AB It is well known that the physicochem. properties of **supercrit.**
fluids (SFs) can be tuned between **gas** and liquid-like
values with only slight changes in temperature and pressure. This tunability
has helped to make SFs attractive **solvents** for use in chemical
reactions, sepsns., and **extraction** techniques. While
supercrit. CO2 (scCO2) is environmentally responsible,
inexpensive, industrially applicable, and the most commonly used SF, it is
a poor **solvent** for polar **solutes**. Reverse micelles
offer a convenient methodol. to enhance the solubility of hydrophiles and
expand the applicability of scCO2. Recently, we and others
demonstrated that one can form stable reverse micelles in scCO2 using a
perfluoropolyether-based surfactant (PFPE) and host hydrophiles as large
as proteins. This presentation will focus on our efforts to determine the
characteristics of the interior water pool within these micelles and their
use as nanobioreactors for enzyme catalysis.
- L98 ANSWER 21 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1997:298833 HCAPLUS
DN 127:56433
ED Entered STN: 10 May 1997
TI On the suitability of the virial equation for modeling the solubility of
solids in **supercritical fluids**
AU Harvey, Allan H.
CS Physical and Chemical Properties Division, Chemical Science and Technology
Laboratory, National Institute of Standards and Technology, Boulder, CO,

USA
SO Fluid Phase Equilibria (1997), 130(1-2), 87-100
CODEN: FPEQDT; ISSN: 0378-3812
PB Elsevier
DT Journal
LA English
CC 68-1 (Phase Equilibria, Chemical Equilibria, and Solutions)
AB Five model systems, the van der Waals fluid, the Soave-Redlich-Kwong fluid, the Peng-Robinson fluid, the hard-sphere fluid, and the square-well fluid, are used to examine the performance of the truncated virial **expansion** in describing the fugacity of a **solute** at infinite dilution in a **solvent**. It is demonstrated that the virial fugacity results deteriorate at significantly lower densities as the **solute** becomes larger. This has consequences for attempts to describe the solubility of solids in **supercrit. fluids**, where the virial **expansion**, truncated after the third virial coefficient, has been considered as a modeling option. The results of this work suggest that, for the densities and **solute-to-solvent** size ratios commonly encountered in **supercrit. extraction**, the truncated virial **expansion** should not be expected to describe correctly the **solute** fugacity, and therefore any success it has in fitting solubility data should be viewed with caution.

ST sly solid **supercrit fluid** virial equation;
solute fugacity **supercrit fluid** virial equation

IT Phase equilibrium
(fluid-solid; suitability of virial equation for modeling solubility of solids in **supercrit. fluids**)

IT Fugacity
Hard-sphere model
Peng-Robinson equation of state
Soave-Redlich-Kwong equation of state
Solubility
Square well potential
Van der Waals equation of state
Virial equation of state
(suitability of virial equation for modeling solubility of solids in **supercrit. fluids**)

IT **Solvents**
(**supercrit.**; suitability of virial equation for modeling solubility of solids in **supercrit. fluids**)

L98 ANSWER 22 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1995:615662 HCAPLUS
DN 123:40911
ED Entered STN: 16 Jun 1995
TI Formation of fine powders of caffeine by RESS
AU Ksibi, H.; Subra, P.; Garrabos, Y.
CS Universite de Paris XIII, Villetaneuse, 93430, Fr.
SO Advanced Powder Technology (1995), 6(1), 25-33
CODEN: APTEEE; ISSN: 0921-8831
PB VSP
DT Journal
LA English
CC 63-8 (Pharmaceuticals)
AB **Precipitation** of solids resulting from solution supersatn. is widely adopted to produce organic and inorg. powders. In fact, the rapid **expansion** of **supercrit. solution** (RESS) is a new process of particle formation. Various morphologies and particle sizes can be thus produced: thin films, thin diameter fibers, needles or spherical products of narrow size distribution. The distinguishing features of this process are the fast attainment of the uniform conditions and of high supersaturations

in the carrier fluid (**supercrit. carbon dioxide**), which favor the formation of small particles, with narrow distribution. The **expansion** of a **supercrit. solution** thus leads to loss of **solvent** power and hence to **solute precipitation**. The RESS is described for the production of fine powders of caffeine from **supercrit. carbon dioxide** upon **expansion**. There is variety of the fluid solution **expansion** parameters. The product morphol., however, can vary considerably depending on the solution components and the operating conditions used in the process: **solute** concentration, preexpansion and **expansion** temperature and pressure of **extraction** have been shown to affect the product characteristics of the formed powder during the process. Optical photomicrographs of the formed particles are compared taking into account the variation of thermodyn. variables. Finally, the variation of the d. distribution and the particle sizes along a plate of deposition is discussed.

ST caffeine powder **supercrit carbon dioxide expansion**

IT Particle size

(production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)

IT Pharmaceutical dosage forms

(powders, production of fine powders by rapid **expansion** of **supercrit. solns.**)

IT 124-38-9, **Carbon dioxide**, uses

RL: NUU (Other use, unclassified); USES (Uses)

(production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)

IT 58-08-2, Caffeine, processes

RL: PEP (Physical, engineering or chemical process); PROC (Process)

(production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)

IT 124-38-9, **Carbon dioxide**, uses

RL: NUU (Other use, unclassified); USES (Uses)

(production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 23 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1994:674375 HCAPLUS

DN 121:274375

ED Entered STN: 10 Dec 1994

TI **Solute** trapping in off-line **supercritical fluid extraction** using controlled modifier condensation.

AU Vejrosta, Jiri; Ansorgova, Alena; Planeta, Josef; Breen, David G.; Bartle, Keith D.; Clifford, Anthony A.

CS Institute of Analytical Chemistry, Academy of Sciences of the Czech Republic, Veveri 97, Brno, 611 42, Czech.

SO Journal of Chromatography, A (1994), 683(2), 407-10
CODEN: JCRAEY; ISSN: 0021-9673

PB Elsevier

DT Journal

LA English

CC 5-1 (Agrochemical Bioregulators)

Section cross-reference(s): 80

AB A new approach to **solvent** trapping, based on controlled modifier condensation, is presented. The trapping system consists of a

fused-silica capillary equipped with a cryofocusing device. As a trapping mechanism, nebulization of **expanding supercrit.** mixture with condensing modifier, followed by analyte trapping into moving liquid layer is assumed. In spiking expts., flufenoxuron was **extracted** with 10% methanol-modified CO₂ and recoveries >90% were found.

The resulting **solvent** vols. needed for quant. trapping are much lower (ca. 0.3 mL) than in the case of direct bubbling through bulk liquid flufenoxuron analytical **supercrit fluid extn**

ST
IT

Extraction

(anal.; **solute** trapping in off-line **supercrit.**

fluid extraction using controlled modifier condensation)

IT

101463-69-8, Flufenoxuron

RL: ANT (Analyte); ANST (Analytical study)

(**solute** trapping in off-line anal. **supercrit.**

fluid extraction using controlled modifier condensation)

L98

ANSWER 24 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN

1994:614317 HCAPLUS

DN

121:214317

ED

Entered STN: 29 Oct 1994

TI

The entropy of hydration of simple hydrophobic **solutes**

AU

Paulaitis, Michael E.; Ashbaugh, Henry S.; Garde, Shekhar

CS

Center for Molecular and Engineering Thermodynamics, Department of Chemical Engineering, University of Delaware, Newark, DE, 19716, USA

SO

Biophysical Chemistry (1994), 51(2-3), 349-57

CODEN: BICIAZ; ISSN: 0301-4622

DT

Journal

LA

English

CC

69-2 (Thermodynamics, Thermochemistry, and Thermal Properties)

AB

Infinite-dilution partial molar entropies of solvation of simple, monoat.

solutes in water are defined in terms of the entropy associated with

(1) **solute** insertion at constant volume and at a fixed position in

the **solvent**, and (2) **expansion** or **contraction**

of the pure **solvent** to maintain constant pressure. A statistical

mech. **expansion** for the entropy of solution in terms of

multiparticle correlation functions is applied to this definition to

identify three intrinsic contributions to the hydration entropy -

solute-solvent pair correlations, rearrangement of

solvent in the vicinity of the **solute** mol., and

expansion or **contraction** of the pure **solvent** -

which the authors evaluate for the inert **gases** in water at

25°C. For the smaller **solutes**, it was found that the

solvent reorganization and **solvent expansion**

contributions offset one another such that the entropy of hydration is

determined almost exclusively by **solute-water** pair correlations. The

solute-water pair correlation entropy also prevails as the primary

factor determining entropies of hydration for the larger **solutes**;

however, **solvent** reorganization now makes a small, neg.

contribution to the entropy.

ST

partial molar entropy hydration hydrophobic **solute**;

solvent solute correlation hydration entropy calcn

IT

Hydration, chemical

(statistical mech. calcn.; entropy of hydration of simple hydrophobic **solutes**)

IT

Solutes

(**hydrophobic**, statistical mech. calcn.; entropy of hydration of simple **hydrophobic solutes**)

IT

Distribution function

(pair correlation, **solute-water**; entropy of hydration of simple hydrophobic **solutes**)

IT

Entropy

(partial molar, of hydration; entropy of hydration of simple hydrophobic **solutes**)

L98 ANSWER 25 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1988:552169 HCAPLUS
DN 109:152169
ED Entered STN: 28 Oct 1988
TI **Supercritical fluid extraction** of
particulate and adsorbent materials. Part 2
AU Wright, B. W.; Smith, R. D.
CS Battelle Pac. Northwest Lab., Richland, WA, USA
SO Report (1987), EPA/600/4-87/040; Order No. PB88-133699, 80 pp. Avail.:
NTIS
From: Gov. Rep. Announce. Index (U. S.) 1988, 88(7), Abstr. No. 817,000
DT Report
LA English
CC 48-1 (Unit Operations and Processes)
Section cross-reference(s): 79, 80
AB The phys. properties of **supercrit. fluids** provide
similar **solvent** strengths as liqs. with higher diffusion
coeffs., lower viscosities, and an extended temperature range which provides
the potential for more rapid and efficient **extraction** rates. The report
describes **expanded** studies for evaluating the applicability and
efficiency of anal. **supercrit.-fluid extraction**
and related methodologies. These studies included the development of
quant., off-line, **supercrit.-fluid extraction**
methodol. and a comparison to traditional Soxhlet **extraction**, the
development and evaluation of online, **supercrit.-fluid**
extraction-gas chromatog. for combined sample preparation and
anal., and direct **supercrit.-fluid extn**
.-mass spectrometry for the monitoring of specific **extraction**
profiles as a function of time. The sample matrixes included an air
particulate sample and XAD-2 resin, polyurethane foam, and Sphero carb
adsorbents that were spiked with various model compds. **CO2**,
isobutane, and MeOH-modified (20 mol %) **CO2** were used as
supercrit. fluids. Related studies on the evaluation of
the quant. anal. capability of a fluorescence-detection, **supercrit**
.-**fluid** chromatog. method and the development of viable
solute focusing methods for capillary **supercrit.-**
fluid chromatog. were conducted.
ST adsorbent **extn supercrit fluid**; analysis
extn supercrit fluid; **gas** chromatog
extn supercrit fluid; mass spectrometry
extn supercrit fluid
IT **Extraction**
(by **supercrit. fluid**, of adsorbents and solids)
IT Chromatography, **gas**
Mass spectroscopy
(**extraction** by **supercrit. fluid** in combination
with)
IT Analysis
(**extraction** in, by **supercrit. fluid**)
IT Adsorbents
(**extraction** of, by **supercrit. fluid**)

L98 ANSWER 26 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1982:562235 HCAPLUS
DN 97:162235
ED Entered STN: 12 May 1984
TI Chromatographic study of the thermodynamics of solutions of hydrocarbons
in liquid **crystal solvents**. Evidence for order
disturbance by the **solutes**
AU Klunder, H.; De Ligny, C. L.
CS Lab. Anal. Chem., Univ. Utrecht, Utrecht, 3522 AD, Neth.

- SO Journal of Solution Chemistry (1982), 11(3), 169-88
CODEN: JSLCAG; ISSN: 0095-9782
- DT Journal
- LA English
- CC 22-13 (Physical Organic Chemistry)
- AB Gas chromatog. expts. were carried out in various phases of the
solvents 4-acetoxy-N-(4-methoxybenzylidene)aniline,
dibutoxyazoxybenzene, Li stearate, dihexyloxyazoxybenzene, and
diheptyloxyazoxybenzene. The **solutes** were linear, branched and
cyclic alkanes, and substituted benzenes. Excess enthalpies, entropies,
and free entropies were calculated from net **retention** vols. In the
nematic liquid **crystalline** phases the effect of order disturbances was
significant in H2e and S2e but it was, by enthalpy-entropy compensation,
not demonstrable in .hivin.G2e. Differences in flexibility and degree of
expansion of the **solutes** did not result in significantly
different values of the excess quantities.
- ST chromatog **solute** liq **crystal**; heat mixing
solute liq **crystal**; orientation liq **crystal**
solute
- IT Heat of mixing
(of hydrocarbons with liquid **crystal solvents**,
chromatog. study of)
- IT Chromatography, **gas**
(of **solutes** in liquid **crystals**, order disturbance in
relation to)
- IT Liquid **crystals**
(orientation of, effect of **solutes** on, chromatog. in relation
to)
- IT Alkanes, properties
Hydrocarbons, properties
RL: PRP (Properties)
(thermodn. of solution with liquid **crystal solvents**,
chromatog. study of)
- IT Entropy
Free energy
(excess, of hydrocarbons with liquid **crystal solvent**,
chromatog. study of)
- IT 2587-42-0 2635-26-9 4485-12-5 10484-13-6 17051-01-3
RL: PRP (Properties)
(orientation of, effect of **solutes** on, **gas**
chromatog. in relation to)
- IT 540-84-1 541-73-1 560-21-4 563-16-6 583-48-2 589-43-5 590-73-8
592-27-8 609-26-7 619-99-8 624-29-3 638-04-0 1067-20-5
1071-26-7 2207-03-6 2207-04-7 2213-23-2 2216-33-3 3221-61-2
3522-94-9 4032-86-4 6876-23-9 15869-80-4 16747-26-5 95-47-6,
uses and miscellaneous 95-49-8 95-50-1 106-42-3, uses and
miscellaneous 106-43-4 106-46-7 108-38-3, uses and miscellaneous
108-41-8 111-65-9, uses and miscellaneous
RL: PROC (Process)
(solns. in liquid **crystal solvents**, chromatog. of)
- IT 98-06-6 104-51-8 111-84-2 124-18-5 493-01-6 493-02-7 620-14-4
622-96-8 871-83-0 922-28-1 926-82-9 1069-53-0 2207-01-4
2216-30-0 2216-34-4 15869-87-1
RL: PROC (Process)
(solns. with liquid **crystal solvents**, chromatog. of)
- L98 ANSWER 27 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
- AN 1979:175379 HCAPLUS
- DN 90:175379
- ED Entered STN: 12 May 1984
- TI Solid solubilities of heavy hydrocarbons in **supercritical**
solvents
- AU Mackay, Michael E.; Paulaitis, Michael E.

CS Dep. Chem. Eng., Univ. Delaware, Newark, DE, USA
 SO Industrial & Engineering Chemistry Fundamentals (1979), 18(2), 149-53
 CODEN: IECFA7; ISSN: 0019-7874
 DT Journal
 LA English
 CC 68-1 (Phase Equilibriums, Chemical Equilibriums, and Solutions)
 Section cross-reference(s): 51
 AB A method is presented for calculating the solubility of condensed, nonvolatile components in **supercrit. solvents** by treating the **supercrit. fluid-phase** mixture as an **expanded liquid**. The procedure is directly applicable to phase equilibrium calcns. associated with **extraction** processes utilizing **supercrit. solvents**. Two mixture parameters are required in the formulation for a binary system-an activity coefficient at infinite dilution for the heavy **solute** and a binary interaction parameter (i.e. k_{12} in the Redlich-Kwong equation of state). The advantage of this approach is that both mixture parameters exhibit consistent, predictable behavior for highly asym. mixts. in the vicinity of the critical region. The utility of this method is illustrated using exptl. data for the solubility of solid naphthalene in **supercrit. carbon dioxide** and in **supercrit. ethylene**.
 ST hydrocarbon soly **supercrit solvent** calcn; heavy hydrocarbon soly **supercrit solvent**; **carbon dioxide supercrit** dissoln hydrocarbon
 IT Solubility
 (calcn. of, for heavy hydrocarbon solids in **supercrit. solvents**)
 IT Hydrocarbons, properties
 RL: PRP (Properties)
 (solubility of solid, in **supercrit. solvents**)
 IT 124-38-9, properties
 RL: PRP (Properties)
 (solubility in **supercrit.**, of heavy hydrocarbon solids)
 IT 124-38-9, properties
 RL: PRP (Properties)
 (solubility in **supercrit.**, of heavy hydrocarbon solids)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

=> d 197 bib abs hitstr retable tot

L97 ANSWER 1 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 2003:950310 HCAPLUS
 DN 140:6706
 TI Electrostatic deposition of particles generated from rapid **expansion of supercritical** fluid solutions
 IN Fulton, John L.; Deverman, George
 PA Battelle Memorial Institute, USA
 SO U.S. Pat. Appl. Publ., 12 pp.
 CODEN: USXXCO
 DT Patent
 LA English
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2003222019	A1	20031204	US 2002-157626	20020528

PRAI US 2002-157626 20020528
AB A method is described for depositing a substance on a substrate, comprising forming a **supercrit.** solution of ≥ 1 **supercrit. solvent** and ≥ 1 **solute**, discharging the **supercrit.** solution through an orifice under conditions sufficient to form solid nanoparticles of the **solute** substantially free of the **supercrit. solvent**, and electrostatically depositing the nanoparticles onto the substrate. The nanoparticles may be charged to a first elec. potential and then deposited onto the substrate to form a film. The **solute** particles have a mean size of $< 1 \mu\text{m}$.
IT 124-38-9, Carbon dioxide, processes
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses) (electrostatic deposition of particles from rapid **expansion** of **supercrit.** solns.)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 2 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2003:950308 HCAPLUS
DN 140:6704
TI Electrostatic deposition of particles from rapid **expansion** of **supercritical** fluid solutions
IN Fulton, John L.; Deverman, George
PA Battelle Memorial Institute, USA
SO U.S. Pat. Appl. Publ., 13 pp.
CODEN: USXXCO
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2003222017	A1	20031204	US 2002-156970	20020528
PRAI	US 2002-156970		20020528		

AB A method is described for depositing a substance on a substrate comprising forming a **supercrit.** solution of ≥ 1 **supercrit. solvent** and ≥ 1 **solute**, discharging the **supercrit.** solution through an orifice under conditions sufficient to form solid nanoparticles of the **solute** free of the **supercrit. solvent**, and electrostatically depositing the nanoparticles onto the substrate. The nanoparticles may be charged to a first elec. potential and then deposited onto the substrate to form a film. The **solute** particles have a mean size of $< 1 \mu\text{m}$.
IT 124-38-9, Carbon dioxide, processes
7440-37-1, Argon, processes 7440-63-3, Xenon, processes
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses) (electrostatic deposition of particles from rapid **expansion** of **supercrit.** solns.)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RN 7440-37-1 HCAPLUS

CN Argon (8CI, 9CI) (CA INDEX NAME)

Ar

RN 7440-63-3 HCAPLUS

CN Xenon (8CI, 9CI) (CA INDEX NAME)

Xe

L97 ANSWER 3 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:915254 HCAPLUS

DN 139:399673

TI Behavior of poly(methyl methacrylate)-based systems in **supercritical CO₂** and **CO₂** plus

cosolvent: Solubility measurements and process assessment

AU Domingo, C.; Vega, A.; Fanovich, M. A.; Elvira, C.; Subra, P.

CS Instituto de Ciencia de Materiales de Barcelona, CSIC, Bellaterra, 08193, Spain

SO Journal of Applied Polymer Science (2003), 90(13), 3652-3659

CODEN: JAPNAB; ISSN: 0021-8995

PB John Wiley & Sons, Inc.

DT Journal

LA English

AB Microspheres based on synthetic polymers such as poly(Me methacrylate) (PMMA) and PMMA blends are known for their medical and optical applications. The development of methods for processing polymeric microspheres using a nontoxic **solvent**, like **supercrit. carbon dioxide** (SCCO₂), is desirable. This work investigates the solubility and behavior of polymers (PMMA and PMMA/polycaprolactone blend) and **solutes** (cholesterol and albumin) in SCCO₂ and SCCO₂ + **cosolvent** (acetone, ethanol, and methylene chloride). The knowledge of solubility behavior of materials in SCCO₂ aids in the selection and/or design of the most appropriate technique for materials processing. Processing PMMA-based polymers with pure SCCO₂ leads to polymer swelling. The lack of polymer solubility in pure **CO₂** precludes their micronization by the RESS (rapid **expansion of supercrit. solns.**) process, but on the other hand allows their impregnation. Polymer plasticization caused by **CO₂** can be exploited in the PGSS (particles from **gas** -saturated solns.) process. Addition of a liquid **cosolvent** to **CO₂** enhances the dissoln. of **solutes** and polymers. **Precipitation** of the studied polymers by **antisolvent** techniques seems feasible only by use of **CO₂** + methylene chloride.

IT 124-38-9, **Carbon dioxide**, processes

RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)

(behavior of poly(Me methacrylate)-based systems in **supercrit. CO₂** and **CO₂** plus **cosolvent**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
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Abraham, G	2000	282	44	J Macromol Mater Eng	HCAPLUS
Alessi, P	1998			Proceedings of the 5	
Berens, A	1992	46	231	J Appl Polym Sci	HCAPLUS
Burkoth, A	2000	21	2389	Biomaterials	
Castellani, S	1996			Ph D Thesis, Univers	
Chang, C	1997	131	243	Fluid Phase Equilib	HCAPLUS
Condo, P	1994	325	23	J Polym Sci Part B:	
Cooper, A	2000	10	207	J Mater Chem	HCAPLUS
Debenedetti, P	1993	24	27	J Controlled Release	HCAPLUS
Domb, A	1994			Polymeric Site Speci	
Domingo, C	1997	10	39	J Supercrit Fluids	HCAPLUS
Domingo, C	2001	21	147	J Supercrit Fluids	HCAPLUS
Du, J	1997	43	223	J Controlled Release	
Engwicht, A	2000	21	1587	Biomaterials	HCAPLUS
Ghaderi, R	1999	16	676	J Pharm Res	HCAPLUS
Hubbell, D	1977	21	3035	J Appl Polym Sci	HCAPLUS
Kim, H	1997	18	1175	Biomaterials	HCAPLUS
Kosal, E	1992	5	169	J Supercrit Fluids	HCAPLUS
Ksibi, H	1996	7	21	Adv Powder Technol	HCAPLUS
Lee, S	1997	38	1317	Polymer	HCAPLUS
Lin, W	2002	198	109	J Membr Sci	HCAPLUS
Liu, G	1996	9	152	J Supercrit Fluids	HCAPLUS
Lucien, F	2000	17	111	J Supercrit Fluids	HCAPLUS
Magnan, C	1996		509	High Pressure Chem E	HCAPLUS
McHugh, M	1994			Supercritical Fluid	
Middleton, J	2000	21	2335	Biomaterials	HCAPLUS
Mishima, K	2000	46	857	AIChE J	HCAPLUS
Reverchon, E	2000	18	239	J Supercrit Fluids	
Robinson, J	1987			Controlled Drug Deli	
Shieh, Y	1996	59	695	J Appl Polym Sci	HCAPLUS
Shieh, Y	1996	59	707	J Appl Polym Sci	HCAPLUS
Shine, A	1997			WO 9815348	HCAPLUS
Siakumar, M	2000	46	29	React Funct Polym	
Siripurapu, S	2000	629	FF991	J Mater Res Soc Symp	
Subra, P	1997	131	269	Fluid Phase Equilib	HCAPLUS
Subra, P	1998	12	261	J Supercrit Fluids	HCAPLUS
Suzuki, K	1990	35	63	J Chem Eng Data	HCAPLUS
Tams, J	1995	16	1049	J Biomaterials	
Thiering, R	2000	75	42	J Chem Technol Biote	HCAPLUS
Vega-Gonzalez, A				J Chem Eng, to appea	
Vincent, M	1997	43	1838	AIChE J	HCAPLUS
Walenkamp, G	1998			Biomaterials in Surg	
West, B	1998	69	911	J Appl Polym Sci	HCAPLUS
Wissinger, R	1987	25	2497	J Polym Sci Part B:	HCAPLUS
Wong, J	1986	2	29	Biotechnol Prog	HCAPLUS
Yun, S	1991	30	2476	Ind Eng Chem Res	HCAPLUS

L97 ANSWER 4 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:843045 HCAPLUS

DN 140:28251

TI Relationship between volume **expansion**, **solvent-power**,
and **precipitation** in **GAS** processes

AU Striolo, Alberto; Elvassore, Nicola; Parton, Tiziana; Bertucco, Alberto

CS Dipt. di Principi e Impianti di Ingegneria Chimica, Universita di Padova,
Padua, I-35131, Italy

SO AIChE Journal (2003), 49(10), 2671-2679

CODEN: AICEAC; ISSN: 0001-1541

PB American Institute of Chemical Engineers

DT Journal

LA English

AB Dilute solns. of Et cellulose (ETC) in acetone and of poly(ethylene oxide)
(PEO) in Et acetate, acetonitrile, Et acetate-acetonitrile, and

acetonitrile - water mixts. were **expanded** isothermally by compressed CO₂. Onset **precipitation** pressures were visually measured through a windowed cell. Toward a rational understanding of the mol. mechanisms involved in **gas antisolvent** (**GAS**) processes, saturated-liquid-phase volume **expansion** and **solvent** power were monitored by UV-vis spectroscopy for the **solvent** mixts. considered in the **precipitation** expts. Ferrocene absorbance and phenol blue absorption-peak-wavelength shifts were used as probes to assess saturated-liquid-phase volume **expansion** and **solvent** power, resp. For the first time, a correlation between a microscopic bulk property, **solvent** power, and the onset **precipitation** pressure of a **solute** is reported. Because of preferential interactions with the dye (hydrogen bonds), the correlation breaks down when even small amts. of water are present in the **solvent** mixture. The results presented here suggest that UV-vis spectroscopy constitutes a valuable tool for understanding some phenomena related to **supercrit.**-fluid technol.

IT 124-38-9, Carbon dioxide, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (relationship between volume **expansion**, **solvent**
 -power, and **precipitation** in **gas antisolvent**
 processes)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Baggio, M	1998			Tesi di Laurea, Univ	
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bertucco, A	1999		231	Proc Int Meeting of	
Brennecke, J	2000			Proc Int Symp Superc	
Carlier, C	1993	39	876	AIChE J	HCAPLUS
Chang, C	1990	36	939	AIChE J	HCAPLUS
Day, C	1996	41	839	J Chem Eng Data	HCAPLUS
De la Fuente Badilla, J	2000	17	13	J Supercrit Fluids	HCAPLUS
Debenedetti, P	1987	42	2203	Chem Eng Sci	HCAPLUS
Debenedetti, P	1989	90	4528	J Chem Phys	HCAPLUS
Eberhardt, R	1997		1195	Liebigs Ann/Recueil	HCAPLUS
Eckert, C	1983	14	167	Fluid Phase Equilib	HCAPLUS
Eckert, C	1986	86	2738	J Phys Chem	
Elvassore, N	2002	42	223	J Chem Eng Data	
Elvassore, N	2001	90	1628	J Pharm Sci	HCAPLUS
Favari, F	2000	55	2379	Chem Eng Sci	HCAPLUS
Figueras, J	1971	93	3255	J Amer Chem Soc	HCAPLUS
Gallagher, P	1989	406		Amer Chem Soc Symp S	HCAPLUS
Kelley, S	1996	42	7	AIChE J	
Kim, S	1987	33	1603	AIChE J	HCAPLUS
Kim, S	1987	26	1206	Ind Eng Chem Res	HCAPLUS
Kolling, O	1973	45	160	Anal Chem	HCAPLUS
Kolling, O	1991	95	3950	J Phys Chem	HCAPLUS
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Morley, J	1999	103	11442	J Phys Chem A	HCAPLUS
Phillips, D	1993	32	943	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Sastri, V	1972	94	753	J Amer Chem Soc	HCAPLUS
Spilimbergo, S	2001	22	55	J Supercrit Fluids	
Subra, P	2000		921	Proc Meeting on Supe	

Subramaniam, B	1997	86	885	J Pharm Sci	HCAPLUS
Sun, Y	1992	114	1187	J Amer Chem Soc	HCAPLUS
Teja, A	2000	39	4442	Ind Eng Chem Res	HCAPLUS
Winters, M	1999	62	247	Biotechnol Bioeng	HCAPLUS
Yamaguchi, T	1993	109	9075	J Chem Phys	

L97 ANSWER 5 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:295462 HCAPLUS

DN 138:306024

TI Vapor-Liquid Mass Transfer during **Gas Antisolvent**

Recrystallization: Modeling and Experiments

AU Lin, Cheng; Muhrer, Gerhard; Mazzotti, Marco; Subramaniam, Bala

CS Institute of Process Engineering, ETH Swiss Federal Institute of Technology Zurich, Zurich, CH-8092, Switz.

SO Industrial & Engineering Chemistry Research (2003), 42(10), 2171-2182

CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

LA English

AB In batch **gas antisolvent (GAS)**

recrystn., the gradual addition of **CO2** to a liquid solution containing the **solute** causes the system pressure to rise and the volume of the liquid phase to **expand** substantially, eventually resulting in **solute precipitation**. The **expansion** rate depends on the rate of **antisolvent** addition and on the vapor-liquid mass-transfer rate and detcs. the rate of supersatn. buildup in solution, which ultimately controls the particle formation process. The effect is studied of mass-transfer resistance on volume **expansion**, both theor. by development of a math. model of the mass-transfer phenomena under typical **GAS recrystn.** conditions and exptl. through volume **expansion** expts. (**CO2** in toluene) to assess the role of operating parameters such as stirring rate and aeration mode. A satisfactory agreement between model results and exptl. data is achieved in all cases.

IT 124-38-9, **Carbon dioxide**, uses

RL: NUU (Other use, unclassified); USES (Uses)

(modeling of vapor-liquid mass transfer in **gas antisolvent recrystn.**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Albal, R	1983	27	61	Chem Eng J	HCAPLUS
Berends, E	1996	42	431	AIChE J	HCAPLUS
Berends, E	1994			Ph D Thesis, Technic	
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bird, R	1960			Transport phenomena,	
Bungert, B	1998	37	3208	Ind Eng Chem Res	HCAPLUS
de La Fuente, B	2000	17	13	J Supercrit Fluids	
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Irving, J	1977			National Engineering	
Jung, J	2001	20	179	J Supercrit Fluids	HCAPLUS
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCAPLUS
Kikic, I	1998	37	1577	Ind Eng Chem Res	HCAPLUS
Knaff, G	1987	21	151	Chem Eng Process	HCAPLUS
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS

Lucas, K	1981	53	959	Chem Ing Tech	HCAPLUS
Muhrer, G	2002	41	3566	Ind Eng Chem Res	HCAPLUS
Muhrer, G	2003			J Supercrit Fluids,	
Muhrer, G	2002			Ph D Thesis, ETH Zur	
Muller, M	2000	39	2260	Ind Eng Chem Res	
Nagata, S	1975			Mixing:Principles an	
Ng, H	1978	23	325	J Chem Eng Data	HCAPLUS
Peng, D	1976	15	59	Ind Eng Chem Fundam	HCAPLUS
Phillips, K	1973	51	371	Can J Chem Eng	HCAPLUS
Reid, R	1987			The properties of li	
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Schluter, V	1992	47	2357	Chem Eng Sci	HCAPLUS
Shariati, A	2002	23	195	J Supercrit Fluids	HCAPLUS
Shizimu, K	1998	191	178	J Cryst Growth	
Subramaniam, B	1997	88	885	J Pharm Sci	
Teramoto, M	1974	8	223	Chem Eng J	HCAPLUS
Werling, J	1999	16	167	J Supercrit Fluids	HCAPLUS
Werling, J	2000	18	11	J Supercrit Fluids	HCAPLUS
Wu, H	1995	50	2801	Chem Eng Sci	HCAPLUS

L97 ANSWER 6 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:269883 HCAPLUS

DN 139:87132

TI DELOS process: a **crystallization** technique using compressed fluids. 1. Comparison to the **GAS crystallization** method

AU Ventosa, N.; Sala, S.; Veciana, J.

CS Institut de Ciencia de Materials de Barcelona (CSIC), Campus Universitari de Bellaterra, Cerdanyola, 08193, Spain

SO Journal of Supercritical Fluids (2003), 26(1), 33-45

CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB The depressurization of an **expanded** liquid organic solution (DELOS) **crystallization** technique is a new 1-step process, which uses a compressed fluid (CF) (e.g. CO₂), for the straightforward production of sub-micron- or micron-sized **crystalline** particles. The driving force of a DELOS **crystallization** process is the fast, large and extremely homogeneous temperature decrease experienced by a solution, which contains a

CF, when it is depressurized from a given working pressure to atmospheric pressure. In contrast to other already reported high-pressure **crystallization** techniques (RESS, **GAS**, PCA, PGSS), in a DELOS process the CF behaves as co-solvent over the initial organic solution of the solute to be **crystallized**. Through a DELOS process it is possible to produce fine powders of a compound provided that a system compound/organic solvent/CF' in a liquid 1-phase state is found. To compare DELOS and **gas anti-solvent (GAS)** procedures, 1,4-bis-(n-butylamino)-9,10-anthraquinone was **crystd** from acetone/CO₂' mixts. by both methods. The **crystn** results obtained were analyzed upon the solubility behavior of 1,4-bis-(n-butylamino)-9,10-anthraquinone in acetone/CO₂' mixts. with different composition. It will be seen how important is the knowledge of the solute solubility behavior in the **CO₂-expanded solvent** to choose the most convenient **crystallization** technique (**GAS** like or DELOS) and the best operational parameters. Finally, it was exptl. determined which are the operational parameters that control the temperature decrease experienced in a DELOS **crystallization**. The results obtained were corroborated through thermodyn. considerations.

IT 124-38-9, Carbon dioxide, processes

RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses)

(comparison of depressurization (DELOS) process to **antisolvent (GAS) crystallization** methods of **crystallization** techniques using compressed fluids.)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Berends, E	1996	42	431	AIChE J	HCAPLUS
Bleich, J	1993	97	111	Int J Pharm	HCAPLUS
Bleich, J	1996	13	131	J Microencapsulation	HCAPLUS
Chang, C	1989	35	1876	AIChE J	HCAPLUS
Dixon, D	1993	50	1929	J Appl Polym Sci	HCAPLUS
Gallagher, P	1989	406	334	ACS Symposium Series	HCAPLUS
Gallagher, P	1992	5	130	J Supercritical Flui	HCAPLUS
Giacobbe, F	1992	72	277	Fluid Phase Equilibr	HCAPLUS
Jung, J	2001	20	179	J Supercrit Fluids	HCAPLUS
Kato, M	1991	24	767	Chem Eng Jap	HCAPLUS
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCAPLUS
Matson, D	1987	26	2298	Ind Eng Chem Res	HCAPLUS
Mawson, S	1997	13	1519	Langmuir	HCAPLUS
Mawson, S	1997	30	71	Macromolecules	HCAPLUS
Mohamed, R	1992	38	742	AIChE J	
Palakodaty, S	1998	1	275	Proceedings of the F	
Randolph, T	1993	9	429	Biotechnol Prog	HCAPLUS
Reverchon, E	1997		335	Proceedings of the F	
SYSTAT inc	1992			SYSTAT for windows,	
Shariati, A	2001		329	Proceedings of the S	
Tom, J	1991	7	403	Biotechnol Prog	HCAPLUS
Ventosa, N	2000			ES 01/00327	
Ventosa, N	2001	1	299	Crystal Growth and D	HCAPLUS
Weidner, E	1994	3	229	Proceedings of the T	
Yeo, S	1993	41	341	Biotechnol Bioeng	HCAPLUS

L97 ANSWER 7 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:182179 HCAPLUS

TI A green process to generate microparticles and nanoparticles

AU Sievers, Robert E.; Quinn, B. P.; Huang, Edward T. S.; Cape, S. P.; Alargov, D. K.; Villa, Joseph A.; Rinner, L.; Meresman, Helena V.; Mitchell, T. L., III; Vander Linden, B. J.

CS CIRES, Department of Chemistry and Biochemistry, and Center for Pharmaceutical Biochemistry, University of Colorado, Boulder, CO, 80309-0215, USA

SO Abstracts of Papers, 225th ACS National Meeting, New Orleans, LA, United States, March 23-27, 2003 (2003), ENVR-107 Publisher: American Chemical Society, Washington, D. C.
CODEN: 69DSA4

DT Conference; Meeting Abstract

LA English

AB Efficient methods for generating fine aerosols are very important for **coating** processes, thin film deposition, fine powder generation and pulmonary drug delivery. Traditionally, aerosols have been generated using liquid **solvents** containing environmentally objectionable organic compds. The process byproducts are toxic organic **solvents** and VOC **gases**. This paper describes a new green process for micronization and nanonization, **Carbon Dioxide-Assisted Nebulization** with a Bubble Dryer-, in which **carbon dioxide** is an

aerosolization agent and water is the **solvent** of choice. Aerosol is generated by intimately mixing dense **carbon dioxide** and an aqueous solution containing a **solute** of interest in a small volume tee at about 83 bar and room temperature. The resultant mixture

is

expanded through a flow restrictor to form an aerosol, which is rapidly dried with **gaseous** nitrogen or air at 30 to 65°C to produce fine dry powders with diams. ranging from about 70 nm to 5 µm. Example of substances from which fine powders have been generated are anti-CD4 monoclonal antibody, α 1-antitrypsin, doxycycline, amoxicillin, tobramycin sulfate, cromolyn sodium, albuterol sulfate, myo-inositol, ovalbumin, lactate dehydrogenase, trypsinogen, lysozyme, trehalose, sucrose, mannitol, potassium chloride and sodium chloride. The authors acknowledge the support of the Colorado Tobacco Research Program (Award No.1R-031) and NIH Leadership Training in Pharmaceutical Biotechnol. (HHS NIGMS Award Number 5 T32 GM08732-02).

L97 ANSWER 8 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:159447 HCAPLUS

DN 138:355605

TI Partial molar volume reduction of **solvent** for **solute** **crystallization** using **carbon dioxide** as **antisolvent**

AU Mukhopadhyay, Mamata

CS Department of Chemical Engineering, I.I.T. Bombay, Bombay, India

SO Journal of Supercritical Fluids (2003), 25(3), 213-223

CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB The **gas antisolvent crystallization** (GASC) process using dense **carbon dioxide** (CO₂) as **antisolvent** is particularly useful for purification and micronization of thermo-labile bioactive solid substances. Conventionally, the GASC process is characterized by the relative total volume **expansion** or the relative molar volume **expansion** of the solution. A new criterion is proposed in this work in terms of the relative partial molar volume reduction (RPMVR) of the **solvent** for selection of the **solvent** and the optimum process condition for the GASC process, as it directly gives a measure of the fraction of the dissolved **solute crystallized**. The **solute** solubility is proportional to the partial molar volume of the **solvent**, v_2 which drastically decreases at a high CO₂ dissoln. This is attributed to clustering of CO₂ mols. around the **solvent** mols. causing the loss of **solvent** power. This results in the desired **antisolvent** effect for lowering the **solute** solubility. v_2 has been calculated for a large number of **solvent-CO₂** liquid mixts. using the Peng-Robinson equation of state. It has been observed that v_2 drastically reduces at a high value of x_1 , irresp. of the fact whether the **solvent** d. is higher or lower than that of the CO₂. The **solute** solubility has been predicted from its value at the ambient pressure and the ratio of the partial molar volumes of the **solvent** with and without CO₂ dissolved in it. The predicted solubility of β -carotene in Et acetate with variation of x_1 at 298 K has been found to compare well with the exptl. observed trend of the GASC process.

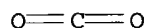
IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)

(partial molar volume reduction of **solvent** for **solute** **crystallization** by using **carbon dioxide** as **antisolvent**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Badilla, J	2000	17	13	J Supercrit Fluids	
Chang, C	1990	36	939	AIChE J	HCAPLUS
Cocero, M	2000			Proceedings of the F	
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Mukhopadhyay, M	2000		50	Natural Extracts Usi	
Mukhopadhyay, M	2001			Proceedings of the 1	
Singh, S	2001			M Tech Dissertation,	

L97 ANSWER 9 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2002:453918 HCAPLUS

DN 137:146138

TI Measurements and modeling of the phase behavior of ternary systems of interest for the **GAS** process: I. The system **carbon dioxide** + 1-propanol + salicylic acid

AU Shariati, A.; Peters, C. J.

CS Faculty of Applied Sciences, Laboratory of Applied Thermodynamics and Phase Equilibria, Delft University of Technology, Delft, 2628 BL, Neth.

SO Journal of Supercritical Fluids (2002), 23(3), 195-208

CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB As a representative model system for the **gas-antisolvent** (**GAS**) process, the phase behavior of the ternary system **carbon dioxide** + 1-propanol + salicylic acid has been studied exptl. For this purpose, **carbon dioxide** has been chosen as the anti-solvent **gas**, 1-propanol as the organic **solvent**, and salicylic acid as the model drug. In each experiment, a solution of salicylic acid in 1-propanol was **expanded** using **carbon dioxide** as the anti-solvent. A synthetic method was used for measuring bubble point curves, and the solid (salicylic acid)-liquid boundaries. Three-phase equilibrium data solid (salicylic acid)-liquid-vapor were obtained from intersection of two-phase isopleths vapor-liquid and solid-liquid. Results are reported for this ternary system at **carbon dioxide** concns. ranging from 8.0 to 90.6 mol%, and within temperature and pressure ranges of 273-367 K and 1.0-12.5 MPa, resp. It has been observed that the **carbon dioxide** concentration significantly affects the optimum operational conditions of the **GAS** process, i.e. at lower concns. **carbon dioxide** acts as a co-solvent, while at higher concns. it acts as an anti-solvent. Also, it is shown that at a proper temperature, it is possible to **precipitate** most of the dissolved **solute** with only a small change of the pressure. The Peng-Robinson equation of state as modified by Stryjek and Vera (PRSV EOS) has been used to model the ternary system.

IT 124-38-9, **Carbon dioxide**, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)

(high-pressure phase equilibrium in **carbon dioxide** /1-propanol/salicylic acid ternary mixts.)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Chang, C	1990	36	939	AIChE J	HCAPLUS
Daubert, T	1989			Physical and Thermod	
De Fina, K	1999	44	1262	J Chem Eng Data	HCAPLUS
de la Fuente Badilla, J	2000	17	13	J Supercrit Fluids	HCAPLUS
Gauter, K	2000	171	127	Fluid Phase Equilib	HCAPLUS
Hanna, M	1997		325	Proceedings of the F	
Jaarmo, S	1997		263	Proceedings of the F	
King, M	1969			Phase Equilibrium in	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Liu, Z	2000	18	111	J Supercrit Fluids	HCAPLUS
Peng, D	1976	15	59	Ind Eng Chem Fund	HCAPLUS
Peters, C	1999	99	419	Chem Rev	HCAPLUS
Peters, C	1987	34	287	Fluid Phase Equilib	HCAPLUS
Peters, C	1993	85	301	Fluid Phase Equilib	HCAPLUS
Prausnitz, J	1986			Molecular Thermodyna	
Reverchon, E	2000	17	239	J Supercrit Fluids	HCAPLUS
Reverchon, E	1999	106	23	Powder Technol	HCAPLUS
Stephen, H	1963			Solubilities of Inor	
Stryjek, R	1986	64	820	Can J Chem Eng	HCAPLUS
Tavana, A	1991	284	5	AIChE Symposium Seri	
Thiering, R	2000	75	29	J Chem Technol Biote	HCAPLUS
Winters, M	1996	85	586	J Pharm Sci	HCAPLUS
Yeo, S	1993	41	341	Biotechnol Bioeng	HCAPLUS

L97 ANSWER 10 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:854939 HCAPLUS

DN 136:119925

TI Formation of Perfluoropolyether **Coatings** by the Rapid
Expansion of Supercritical Solutions (RESS) Process.
 Part 2: Numerical Modeling

AU Franklin, Randall K.; Edwards, Jack R.; Chernyak, Yury; Gould, Richard D.;
 Henon, Florence; Carbonell, Ruben G.

CS Department of Mechanical and Aerospace Engineering and Department of
 Chemical Engineering, North Carolina State University, Raleigh, NC, 27695,
 USA

SO Industrial & Engineering Chemistry Research (2001), 40(26), 6127-6139
 CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

LA English

AB The rapid **expansion of supercrit.** solns. (RESS)
 process is a promising method for the production of ultrafine powders and
 aerosols of narrow size distribution for **coatings** and other
 applications. In this article, part 2 of a two-part study, the nucleation
 and subsequent growth of 2500 Mw perfluoropolyether diamide (PFD) from
supercrit. carbon dioxide (CO2) by
expansion through a small-diameter nozzle is modeled in a
 three-stage, multidimensional fashion. The stages include a hydrodynamic
 solution, **solvent-solute** phase equilibrium analyses, and an
 aerosol transport model. The hydrodynamics model successfully captures
 the vapor-liquid transition that occurs as **carbon dioxide**
 is **expanded** to ambient conditions. Cloud-point pressures and
 equilibrium compns. of the separated **solvent-solute** system are
 determined and are used in a multidimensional aerosol transport model. This
 model incorporates various mechanisms influencing droplet growth.

Parametric studies are conducted to investigate the influences of the interfacial tension, the equilibrium addition of **carbon dioxide**, and the diffusion coefficient on the predicted droplet diameter. Turbulent coagulation in the ambient region downstream of the **expansion** nozzle is found to be the dominant mechanism responsible for the production of micron-sized droplets observed in companion expts.

IT 124-38-9, **Carbon dioxide**, uses

RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)

(formation of perfluoropolyether **coatings** by rapid **expansion** of **supercrit.** solns. (RESS) process. part 2: numerical modeling)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Chernyak, Y	2002	41	xxxxx	Ind Eng Chem Res	
Chernyak, Y	2000			Proceedings of the 5	
Debenedetti, P	1993	82	311	Fluid Phase Equilib	HCAPLUS
Edwards, J	2000	38	1624	AIAA J	HCAPLUS
Franklin, R	2000			Master's Thesis, Nor	
Friedlander, S	1977			Smoke, Dust, and Haz	
Hannay, J	1879	30	178	Proc R Soc London	
Harrison, K	1998	14	6855	Langmuir	HCAPLUS
Jung, J	2001	20	179	J Supercrit Fluids	HCAPLUS
Krukoni, V	1984			Presented at the AIC	
Ksibi, H	1996	10	69	Chem Biochem Eng Q	HCAPLUS
Kumar, S	1988	1	15	J Supercrit Fluids	HCAPLUS
Kwauk, X	1993	24	445	J Aerosol Sci	HCAPLUS
Lele, A	1992	38	742	AIChE J	HCAPLUS
Lindsay, J	1999			M S Thesis, North Ca	
Matson, D	1987	26	2298	Ind Eng Chem Res	HCAPLUS
Mawson, S	1995	28	3182	Macromolecules	HCAPLUS
McBride, B	1993			Coefficients for Cal	
Olchowny, G	1988	61	15	Phys Rev Lett	
Prausnitz, J	1986			Molecular Thermodyna	
Saffman, P	1956	1	16	J Fluid Mech	
Sanchez, I	1976	80	2352	J Phys Chem	HCAPLUS
Sanchez, I	1994		187	Models for Thermodyn	
Schaaf, P	1987	28	1930	Polymer	
Spalart, P	1992	1	5	Rech Aerosp	
Span, R	1996	25	1511	J Phys Chem Ref Data	
Wilcox, D	1998			Turbulence Modeling	
Wilke, C	1950	18	517	J Chem Phys	HCAPLUS
Zoller, P	1995		255	Standard Pressure-Vo	

L97 ANSWER 11 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:847154 HCAPLUS

DN 136:119892

TI Formation of Perfluoropolyether **Coatings** by the Rapid

Expansion of **Supercritical** Solutions (RESS) Process.

Part 1: Experimental Results

AU Chernyak, Yury; Henon, Florence; Harris, Robert B.; Gould, Richard D.; Franklin, Randall K.; Edwards, Jack R.; DeSimone, Joseph M.; Carbonell, Ruben G.

CS Department of Chemical Engineering and Department of Mechanical and

Aerospace Engineering, North Carolina State University, Raleigh, NC,
27695, USA

SO Industrial & Engineering Chemistry Research (2001), 40(26), 6118-6126
CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

LA English

AB The rapid **expansion** of **supercrit.** solns. (RESS)
process is a promising environmentally benign technol. for fine droplet or
particle formation. The absence of organic **solvents** and narrow
size distribution of RESS **ppts.** make this process attractive for
polymer **coating** applications. This technique has been used to
produce droplets of perfluoropolyethers from CO₂ solns. without
the aid of **cosolvents** for the **coating** of porous
materials applied in monumental and civil infrastructures. The present
work is aimed at gaining an understanding of the relationship between
droplet and spray characteristics and RESS process conditions. As such, a
combined exptl./computational approach is applied to a representative
binary system consisting of a low-mol.-weight perfluoropolyether diamide
(PFD) dissolved in **supercrit.** CO₂. Part 1 of this
work presents phase equilibrium measurements and polymer droplet size
characterizations under different operating conditions. The effects of
temperature, **solute** concentration, and nozzle configuration on droplet and
spray characterization and transfer efficiency are discussed. Part 2 of
this work presents a multidimensional computational fluid dynamics model
of the RESS **expansion** process and describes the use of the model
in further analyzing and interpreting exptl. data.

IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)

(**supercrit.**, **solvent**; formation of
perfluoropolyether **coatings** by rapid **expansion** of
supercrit. solns. process)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Brennecke, J	1989	35	1409	AIChE J	HCAPLUS
Castelvetto, V	1998	11	551	Surf Coat Int	
Chang, C	1989	35	1876	AIChE J	HCAPLUS
Debenedetti, P	1990	36	1289	AIChE J	HCAPLUS
Debenedetti, P	1993	82	311	Fluid Phase Equilib	HCAPLUS
Desimone, J	1992	257	945	Science	HCAPLUS
Domingo, C	1997	10	35	J Supercrit Fluids	
Donohue, M	1995		152	Green Chemistry	
Franklin, R	2002	41	xxxxx	Ind Eng Chem Res	
Henon, F	1999			Ph D Thesis, North C	
Kim, J	1996	12	650	Biotechnol Prog	HCAPLUS
Ksibi, H	1996	10	69	Chem Biochem Eng Q	HCAPLUS
Ksibi, H	1994		331	Proceedings of the 3	
Kwauk, X	1993	24	445	J Aerosol Sci	HCAPLUS
Lele, A	1994	33	1476	Ind Eng Chem Res	HCAPLUS
Lewis, J	1997		33	Met Finish	HCAPLUS
Liu, G	1997	30	293	J Chem Eng Jpn	HCAPLUS
Matson, D	1987	26	229	Ind Eng Chem Res	
Mawson, S	1995	28	3182	Macromolecules	HCAPLUS
McHugh, M	1994			Supercritical Fluid	

Mohamed, S	1989	35	325	AIChE J	
Muirhead, J	1974		248	Science and Technolo	
Piacenti, F	1994	68	227	J Fluorine Chem	HCAPLUS
Piacenti, F	1994	143	113	Sci Total Environ	HCAPLUS
Sanchez, I	1976	80	2352	J Phys Chem	HCAPLUS
Sanchez, I	1994		187	Models for Thermodyn	
Schaub, G	1995	8	318	J Supercrit Fluids	
Shim, J	1999	38	3655	Ind Eng Chem Res	HCAPLUS
Sianesi, D	1971	18	85	Wear	HCAPLUS
Span, R	1996	25	1511	J Phys Chem Ref Data	
Tom, J	1991	22	555	J Aerosol Sci	HCAPLUS
Zoller, P	1995		255	Standard Pressure-Vo	

L97 ANSWER 12 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:792800 HCAPLUS

Correction of: 2000:812660

DN 135:322653

Correction of: 134:61420

TI **Supercritical antisolvent** micronization of some biopolymers

AU Reverchon, E.; Della Porta, G.; De Rosa, I.; Subra, P.; Letourneur, D.
CS Dipartimento di Ingegneria Chimica e Alimentare, Universita di Salerno, Fisciano, 84084, Italy

SO Journal of Supercritical Fluids (2000), 18(3), 239-245
CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB We proposed various biopolymers by semi-continuous **supercrit. antisolvent precipitation** (SAS) to evaluate the possibility of producing nano- and microparticles of controlled size and distribution. First, some liquid **expansion** curves were exptl. produced to study the general behavior of the ternary systems **antisolvent-solvent**-biopolymer. A condition that guarantees a successful SAS micronization is that **solute** does not modify the **expansion** curves of the **solvent-antisolvent** binary system. SAS expts. were performed by varying the process parameters; we mainly studied the influence of pressure, temperature and liquid solution concns. SEM images of the processed material were used to study morphologies, mean particle size and particle size distribution. We successfully processed by SAS dextran, poly(L-lactide) and poly(hydroxypropylmethacrylamide) by using DMSO and dichloromethane as liquid **solvents**.

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Benedetti, L	1997	53	232	Biotech Bioeng	HCAPLUS
Benedetti, L	1995		221	Proceedings of the T	
Bertuccio, A	1996		217	Proceedings of High	HCAPLUS
Bleich, J	1993	97	111	Internat J Pharm	HCAPLUS
Bleich, J	1994	106	77	Internat J Pharm	HCAPLUS
Bodmeier, R	1995	13	1211	Pharm Res	
Chou, Y	1997		55	Proceedings of the F	
Dillow, A	1997		247	Proceedings of the F	
Falk, R	1997		109e	Presented at AIChE A	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Mawson, S	1997	64	2105	J Appl Polym Sci	HCAPLUS
Randolph, T	1993	9	429	Biotech Progress	HCAPLUS
Rantakyla, M	1998	1	333	Proceedings of the 5	
Reverchon, E	1998	37	952	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1998	13	284	J Mater Res	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS

Reverchon, E	2000	17	239	J Supercrit Fluids	HCAPLUS
Reverchon, E	1999	102	129	Powder Technol	
Reverchon, E	1999		579	Proceedings of the F	
Saim, S	1996	13	S273	Pharm Res	
Thies, J	1998	45	67	Eur J Pharm Biopharm	HCAPLUS
Yeo, S	1993	26	6207	Macromolecules	HCAPLUS

L97 ANSWER 13 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:670920 HCAPLUS

DN 135:373704

TI Microparticle formation and **crystallization** rate of HMX with **supercritical CO2 antisolvent recrystallization**

AU Cai, Jianguo; Zhou, Zhanyun; Deng, Xiu

CS Chemical Engineering Research Center, East China University of Science and Technology, Shanghai, 200237, Peop. Rep. China

SO Chinese Journal of Chemical Engineering (2001), 9(3), 258-261
CODEN: CJCEEB; ISSN: 1004-9541

PB Chemical Industry Press

DT Journal

LA English

AB Microparticle formation and **crystallization** rate of 1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane (HMX) in acetone solution by using **supercrit. carbon dioxide antisolvent (GAS) recrystn.** were studied. Scanning electronic microscopy, X-ray diffraction and IR radiation were used to examine particle size, **crystallinity** and chemical structure. The β -HMX microparticle in different average size (2-9.5 μ m) and with narrow size distribution were obtained by controlling the **expansibility, expansion** speed, initial concentration and temperature during **recrystn.** of HMX. The formation of nuclei is the a main cause of consumption of **solute** when the solution is **expanded** rapidly enough and the equilibrium concentration is lower, in which almost monodisperse microparticle can be obtained.

IT 124-38-9, Carbon dioxide, uses

RL: PEP (Physical, engineering or chemical process); PRP (Properties); TEM (Technical or engineered material use); PROC (Process); USES (Uses)
(microparticle formation and **crystallization** rate of HMX with **supercrit. CO2 antisolvent recrystn**
.)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Gallagher, P	1989		334	Supercritical Fluid	HCAPLUS
Gibbs, J	1957	1	322	Thermodynamics	
Hannay, J	1879	29	324	Proc Roy Soc	
Hoffsommer, J	1975	103	182	J Chromatography	HCAPLUS
Krukonis, V	1984			Ann Mtg AIChE	
Larsen, K	1986		73	Biotech Prog	
Nielsen, A	1964		350	Kinetic of Precipita	
Nyvlt, J	1971		189	Industrial Crystalli	
Worthy, W	1981		16	C&E	
Yeo, S	1993	41	241	Biotech Bioeng	

L97 ANSWER 14 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:195526 HCAPLUS
 DN 134:210028
 TI Fine particle **coating** for release control by using RESS process in fluidized bed
 AU Wang, Tingjie; Tsutsumi, Atsushi; Jin, Yong
 CS Dep. Chem. Eng., Tsinghua Univ., Beijing, 100084, Peop. Rep. China
 SO Huagong Xuebao (Chinese Edition) (2001), 52(1), 50-55
 CODEN: HUKHAI; ISSN: 0438-1157
 PB Huaxue Gongye Chubanshe, Huagong Xuebao Bianjibu
 DT Journal
 LA Chinese
 AB Fine particle **coating** was conducted by the rapid **expansion** of **supercrit.** fluid solution (RESS) in a fluidized bed for release control of some key component in core particles. The **supercrit. carbon dioxide** solution of paraffin was jetted into the fluidized bed of the core particles. The rapid phase change of the fluid solution from **supercrit.** state to **gas** results in a **solute** at high supersaturating state in the **solvent**, which forms a huge number of superfine nuclei in the jetting flow. The deposition of the superfine nuclei on the surface of the core particles leads to a thin layer **coating** of paraffin. The size of the superfine nuclei is in the order of 40 nm. A porous spherical particle was selected as the core particle, which carried a tracer component of a kind of dye. **Coating** level was examined by the tracer's release concentration in a **solvent** over a certain time. The state of **coating** was analyzed by measuring the average mass of **coated** particles and a SEM observation on the surface of **coated** particles. The rapid **expansion** of the **supercrit.** fluid solution causes a big temperature drop at the nozzle outlet. The low temperature of the nozzle outlet affects the phase of **carbon dioxide** and the properties of the superfine nuclei in the jetting flow, therefore it affects the particle **coating** process. The effect of temperature at nozzle inlet, an important parameter, on surface **coating** was investigated. Seal **coating** was formed on the core particle surface at higher temperature. Porous **coating** was formed on the core particle surface at lower temperature. The temperature of the nozzle inlet affects the nucleus size significantly. Higher temperature results in a bigger size of the superfine nuclei. By controlling the operation parameters, a satisfactory quality of **coated** particles was achieved.
 IT 124-38-9, Carbon dioxide, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (fine particle **coating** for release control by using rapid **expansion** of **supercrit.** fluid soluble process in fluidized bed)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 15 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 2000:812660 HCAPLUS
 DN 134:61420
 TI **Supercritical antisolvent** micronization of some biopolymers
 AU Reverchon, E.; Della Porta, G.; De Rosa, I.; Subra, P.; Letourneur, D.
 CS Dipartimento di Ingegneria Chimica e Alimentare, Universita di Salerno, Fisciano, 84084, Italy
 SO Journal of Supercritical Fluids (2000), 18(3), 239-245
 CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB We proposed various biopolymers by semi-continuous **supercrit. antisolvent precipitation** (SAS) to evaluate the possibility of producing nano- and microparticles of controlled size and distribution. First, some liquid **expansion** curves were exptl. produced to study the general behavior of the ternary systems **antisolvent-solvent**-biopolymer. A condition that guarantees a successful SAS micronization is that **solute** does not modify the **expansion** curves of the **solvent-antisolvent** binary system. SAS expts. were performed by varying the process parameters; we mainly studied the influence of pressure, temperature and liquid solution concns. SEM images of the processed material were used to study morphologies, mean particle size and particle size distribution. We successfully processed by SAS dextran, poly(L-lactide) and poly(hydroxypropylmethacrylamide) by using DMSO and dichloromethane as liquid **solvents**.

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Benedetti, L	1997	53	232	Biotech Bioeng	HCAPLUS
Benedetti, L	1995		221	Proceedings of the T	
Bertucco, A	1996		217	Proceedings of High	HCAPLUS
Bleich, J	1993	97	111	Internat J Pharm	HCAPLUS
Bleich, J	1994	106	77	Internat J Pharm	HCAPLUS
Bodmeier, R	1995	13	1211	Pharm Res	
Chou, Y	1997		55	Proceedings of the F	
Dillow, A	1997		247	Proceedings of the F	
Falk, R	1997		109e	Presented at AIChE A	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Mawson, S	1997	64	2105	J Appl Polym Sci	HCAPLUS
Randolph, T	1993	9	429	Biotech Progress	HCAPLUS
Rantakyla, M	1998	1	333	Proceedings of the 5	
Reverchon, E	1998	37	952	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1998	13	284	J Mater Res	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Reverchon, E	2000	17	239	J Supercrit Fluids	HCAPLUS
Reverchon, E	1999	102	129	Powder Technol	
Reverchon, E	1999		579	Proceedings of the F	
Saim, S	1996	13	S273	Pharm Res	
Thies, J	1998	45	67	Eur J Pharm Biopharm	HCAPLUS
Yeo, S	1993	26	6207	Macromolecules	HCAPLUS

L97 ANSWER 16 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2000:812654 HCAPLUS

DN 133:337029

TI Influence of thermodynamic behaviour and **solute** properties on homogeneous nucleation in **supercritical** solutions

AU Turk, Michael

CS Institut fur Technische Thermodynamik und Kaltetechnik, Universitat Karlsruhe (TH), Karlsruhe, D-76131, Germany

SO Journal of Supercritical Fluids (2000), 18(3), 169-184

CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB The knowledge about the thermodyn. behavior of dilute **supercrit.** solns. is one of the basics for modeling processes, such as the formation of small particles by rapid **expansion** of **supercrit.** solns. (RESS). RESS allows the production of particles less than 1 μ m and RESS expts. show that particle size depends on **solvent**,

solute and preexpansion conditions. However, an understanding of the underlying phys. phenomena of the relationship between the process conditions and the mechanism of particle formation during RESS is still at an early stage. Because of that, there is a need to model the RESS process to get a better understanding of the influencing parameters. The calcns. show a steep increase at the beginning of the freejet reaching maximum theor. supersaturations of ≈ 108 and for an interfacial tension of 0.02 N m^{-1} maximum nucleation rates of about $10+26 \text{ (cm}^{-3} \text{ s}^{-1})$. In the present paper, the influence of the solubility of various **solutes** in **supercrit.** fluids and of the surface tension group ($\sigma \propto \sqrt{S_2/3/k \cdot T}$) of the diverse **solutes** on attainable nucleation rates under typical RESS operation conditions is investigated. The calcns. show that the nucleation rate is a sensitive function of the solubility and of the unknown surface tension group. Furthermore, it is shown that the classical nucleation theory is not able to describe the trend in particle size resulting from RESS expts. in a sufficient manner. Also, the present calcns. show that it is not possible to investigate homogeneous nucleation and coagulation sep. and that there is an enormous need for more reliable information about the **solute** properties.

IT 124-38-9, Carbon dioxide, processes

RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)

(thermodn. behavior and **solute** properties in homogeneous particle nucleation in **supercrit.** solns.)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Abraham, O	1981	75	402	J Chem Phys	HCAPLUS
Cihlar, S	1999	30	355	J Aerosol Sci	
Cihlar, S	1998		3	Proceedings of the W	
Debenedetti, P	1986	32	1253	Am Inst Chem Eng J	HCAPLUS
Debenedetti, P	1990	36	1289	Am Inst Chem Eng J	HCAPLUS
Foster, N	1991	30	1955	Ind Eng Chem Res	HCAPLUS
Helfgen, B	2000	110	22	J Powder Technol	HCAPLUS
Helfgen, B	1998		14	Proceedings of the A	
Jasper, J	1972	1	841	J Phys Chem Ref Data	HCAPLUS
Kodas, T	1986	111	102	J Colloid Interf Sci	HCAPLUS
Kruis, F	1993	19	514	Aerosol Sci Technol	HCAPLUS
Kwauk, X	1993	24	445	J Aerosol Sci	HCAPLUS
Lyman, W	1990			Handbook of Chemical	
Meyer, J	1998	3	31	Proceedings of the W	
Mohamed, R	1989	35	325	Am Inst Chem Eng J	HCAPLUS
Niekrawietz, M	1989			Dissertation, Univer	
Platzer, B	1989	10	223	Fluid Phase Equilib	
Pratsinis, S	1988	124	416	J Colloid Interf Sci	HCAPLUS
Preining, O	1998	29	481	J Aerosol Sci	HCAPLUS
Schmitt, W	1986	31	204	Chem Eng Data	HCAPLUS
Shaub, G	1995	8	318	J Supercrit Fluids	HCAPLUS
Singh, H	1993	32	2841	Ind Eng Chem Res	HCAPLUS
Springer, G	1978	14	281	Adv Heat Transfer	HCAPLUS
Tom, J	1991	22	555	J Aerosol Sci	HCAPLUS
Treffinger, P	1994	7	251	Fortschritt-Berichte	
Turk, M	1993			Dissertation, Univer	
Turk, M	1999	15	79	J Supercrit Fluids	HCAPLUS

Turk, M	1999	235	Proceedings of the I
Vargaftik, N	1996		Handbook of Physical

L97 ANSWER 17 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2000:802758 HCAPLUS

DN 134:136546

TI Synthesis, Purification, and Micronization of Pharmaceuticals Using the
Gas Antisolvent Technique

AU Warwick, B.; Dehghani, F.; Foster, N. R.; Biffin, J. R.; Regtop, H. L.

CS School of Chemical Engineering and Industrial Chemistry, University of New
South Wales, Sydney, 2502, Australia

SO Industrial & Engineering Chemistry Research (2000), 39(12), 4571-4579

CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

LA English

AB The synthesis, purification, and micronization of the nonsteroidal antiinflammatory Cu₂(indomethacin)₄L₂ (Cu-Indo); (L = DMF) has been investigated using DMF as the **solvent** and CO₂ as the **antisolvent**. The phase behavior of the binary system DMF + CO₂ and the ternary system DMF + CO₂ + Cu-Indo at 25, 30, and 40 °C and pressures up to 7.6 MPa was examined. The phase behavior of the ternary system DMF + CO₂ containing copper(II) acetate monohydrate (Cu-Acetate), indomethacin, or acetic acid and the quaternary system DMF + CO₂ containing Cu-Indo and either Cu-Acetate, indomethacin, or acetic acid at 25 °C and pressures up to 5.8 MPa was also examined to determine optimum synthesis conditions. The effect of variables such as reactant concentration, CO₂ wash volume, and rate of **expansion** on the purity and characteristics of the Cu-Indo produced in the synthesis was investigated. The **recrystn** of Cu-Indo from DMF was investigated and the effect of the rate of **expansion** on the size of the particles produced was determined at 25 °C. It was found that Cu-Indo solubility in a DMF **expanded** solution decreased with increasing pressure and decreasing temperature. The solubility

of Cu acetate in a DMF **expanded** solution was slightly increased as the pressure increased to 2.7 MPa and decreased rapidly at higher pressures. Upon addition of CO₂ to DMF + indomethacin saturated solns., a second liquid phase formed in the system and **precipitation** only occurred at pressures above 5.5 MPa. Acetic acid was found to remain soluble in the DMF **expanded** solution at the range of pressures and temps. examined. The addition of a second **solute** to the DMF + CO₂ + Cu-Indo solns. was found to significantly influence the phase behavior of the system. The solubility of Cu-Indo increased in the presence of acetic acid and Cu-Acetate and decreased in the presence of indomethacin. The product, Cu-Indo, with greater than 95% purity was produced in a single step at 25 °C. The presence of a slight excess of either reactant did not alter the purity of the Cu-Indo produced. The rate of **expansion** substantially varied the size and morphol. of the particles produced. Rapid **expansion** resulted in bipyramidal **crystalline** particles that were less than 10 µm in size. Slow **expansion** resulted in rhombic **crystals** with an average size of between 20 and 10 µm.

IT 124-38-9, Carbon dioxide, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties);
PROC (Process)

(preparation, purification, and micronization of pharmaceuticals using the
gas antisolvent technique)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bungert, B	1997	69	298	Chem Ing Tech	HCAPLUS
Bungert, B	1998	37	3208	Ind Eng Chem Res	HCAPLUS
Chang, C	1991	7	275	Biotechnol Prog	HCAPLUS
Chang, C	1994	72	56	Can J Chem Eng	HCAPLUS
Chang, C	1995	40	850	J Chem Eng Data	HCAPLUS
Chang, C	1993	26	517	J Chem Eng Jpn	HCAPLUS
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Foster, N	1997		27	The 4th International	
Gallagher, P	1989	406	334	ACS Symposium Series	HCAPLUS
Griffith, A	1999	38	411	Polym Plast Technol	HCAPLUS
Jianguo, C	1996	4	257	Chin J Chem Eng	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Liou, Y	1992	27	1277	Sep Sci Technol	HCAPLUS
Regtop, H	1990			WO 9014337	HCAPLUS
Regtop, H	1994			US 5310936	HCAPLUS
Regtop, H	1995			US 5466824	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Savage, P	1995	41	1723	AIChE J	HCAPLUS
Shishikura, A	1994	42	1993	J Agric Food Chem	HCAPLUS
Shishikura, A	1997		51	The 4th International	
Sorenson, R	1989			Progress in Medicina	
Subramaniam, B	1986	25	1	Ind Eng Chem Process	HCAPLUS
Tai, C	1998	44	989	AIChE J	HCAPLUS
Thiering, R	2000	75	29	J Chem Technol Biote	HCAPLUS
Weder, J	1999	38	1736	Inorg Chem	HCAPLUS

L97 ANSWER 18 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1999:698745 HCAPLUS

DN 132:266692

TI **Gas antisolvent recrystallization of specialty chemicals**

AU Muhrer, Gerhard; Mazzotti, Marco

CS Institut fur Verfahrenstechnik, ETH Zurich, Zurich, CH-8092, Switz.

SO International Symposium on Industrial Crystallization, 14th, Cambridge, United Kingdom, Sept. 12-16, 1999 (1999), 330-339 Publisher: Institution of Chemical Engineers, Rugby, UK.

CODEN: 68IRAJ

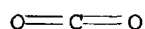
DT Conference; General Review; (computer optical disk)

LA English

AB A review with 84 refs. The need for the manufacturing of micron or sub-micron particles with narrow size distributions is gaining more and more importance in the production of specialty chems. and pharmaceuticals. In the last case microparticles are often intended for controlled drug release applications. There is therefore an increasing interest in developing technologies which, contrary to conventional techniques, allow microparticles with controlled particle size distribution and product quality to be produced under mild and inert conditions. **Supercrit** fluid technol., particularly when using **carbon dioxide**, offers promising possibilities for tackling this challenge, e.g., through the Rapid **Expansion of Supercrit. Solns.**, **Precipitation with Compressed Antisolvent**, and **GAS (Gas Anti-Solvent)** techniques. In particular, **GAS recrystn.** exploits the low solubility of pharmaceutical compds. in **supercrit. carbon dioxide**, which

is used as **antisolvent** for the **solute** initially solubilized in a conventional **solvent**. Upon mixing by adding compressed **carbon dioxide** to the initial solution in a vessel, the solution is **expanded**, thus reducing its **solvent** power, and the **solute** **ppts.** Numerous exptl. investigations have proved the attractiveness of these processes in terms of product quality; however, the understanding of their fundamentals and of the effects of individual process parameters is still very limited. The development of applications of the **GAS recrystn.** technol. requires that the gap between exptl. evidence and theor. understanding is filled.

IT 124-38-9, Carbon dioxide, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (supercrit.; in gas antisolvent
 recrystn. of specialty chems.)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Alessi, P	1996	35	4718	Ind Eng Chem Res	HCAPLUS
Aniedobe, N	1997	30	2792	Macromol	HCAPLUS
Beckmann, W	1997	69	349	Chem Ing Tech	HCAPLUS
Benedetti, L	1997	53	232	Biotechnol Bioeng	HCAPLUS
Berends, E	1996	42	431	AIChE J	HCAPLUS
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bodmeier, R	1995	12	1211	Pharm Res	HCAPLUS
Bungert, B	1997	139	349	Fluid Phase Equilibr	HCAPLUS
Bungert, B	1998	37	3208	Ind Eng Chem Res	HCAPLUS
Catchpole, O	1996	12	309	Process Technology P	HCAPLUS
Chang, C	1989	35	1876	AIChE J	HCAPLUS
Chang, C	1990	36	939	AIChE J	HCAPLUS
Chang, C	1991	7	275	Biotechnol Progress	HCAPLUS
Chang, C	1994	72	56	Can J Chem Eng	HCAPLUS
Chang, C	1993	26	517	J Chem Eng Japan	HCAPLUS
Debenedetti, P	1990	36	1289	AIChE J	HCAPLUS
Debenedetti, P	1993	82	311	Fluid Phase Equilibr	HCAPLUS
Debenedetti, P	1993	24	27	J Controlled Rel	HCAPLUS
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Dixon, D	1993	39	127	AIChE J	HCAPLUS
Dixon, D	1993	50	1929	J Appl Polymer Sci	HCAPLUS
Domingo, C	1996	166	989	J Cryst Growth	HCAPLUS
Domingo, C	1997	10	39	J Supercrit Fluids	HCAPLUS
Falk, R	1997	44	77	J Controlled Rel	HCAPLUS
Falk, R	1998	15	1233	Pharm Res	HCAPLUS
Furuta, S	1995	148	197	J Cryst Growth	HCAPLUS
Gallagher, P	1989	406	334	ACS Symp Ser	HCAPLUS
Gallagher, P	1991	284	96	AIChE Symp Ser	
Gallagher, P	1992	5	130	J Supercrit Fluids	HCAPLUS
Griscik, G	1995	155	112	J Cryst Growth	HCAPLUS
Gromov, D	1998	108	4647	J Chem Phys	HCAPLUS
Gupta, P	1991	17	129	J Controlled Rel	
Jianguo, C	1996	4	257	Chin J Chem Eng	
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCAPLUS
Kim, J	1996	12	650	Biotechnol Progress	HCAPLUS
Kitamura, M	1997	178	378	J Cryst Growth	HCAPLUS
Knutson, B	1996	77	89	Drugs and the pharma	HCAPLUS

Kohn, J	1994	21	132	Proceed Intern Symp	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Kwauk, X	1993	24	445	J Aerosol Sci	HCAPLUS
Larson, K	1986	2	73	Biotechnol Progress	HCAPLUS
Lele, A	1992	38	742	AIChE J	HCAPLUS
Lele, A	1994	33	1476	Ind Eng Chem Res	HCAPLUS
Liou, Y	1992	27	1277	Sep Sci Technol	HCAPLUS
Luna-Barcenas, G	1998	146	325	Fluid Phase Equilibr	HCAPLUS
Luna-Barcenas, G	1995	36	3173	Polymer	HCAPLUS
Matson, D	1986	1	242	Adv Ceram Mater	HCAPLUS
Matson, D	1987	21	109	Adv in Ceram	HCAPLUS
Mawson, S	1997	64	2105	J Appl Polymer Sci	HCAPLUS
Mawson, S	1997	13	1519	Langmuir	HCAPLUS
Mawson, S	1995	28	3182	Macromol	HCAPLUS
Mawson, S	1997	30	71	Macromol	HCAPLUS
Mawson, S	1997	38	2957	Polymer	HCAPLUS
Mishima, K	1998	61	179	Fukuoka University R	HCAPLUS
Mohamed, R	1989	406	355	ACS Symp Ser	HCAPLUS
Niehaus, M	1997	22	176	Prop Expl Pyrotech	HCAPLUS
Ohgaki, K	1990	3	103	J Supercrit Fluids	HCAPLUS
Reverchon, E	1998	37	952	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1998	13	284	J Mater Res	HCAPLUS
Reverchon, E	1993	6	241	J Supercrit Fluids	HCAPLUS
Reverchon, E	1996	9	216	J Supercrit Fluids	HCAPLUS
Reverchon, E	1998	118	349	Stud Surf Sci Catal	HCAPLUS
Schmitt, W	1995	41	2476	AIChE J	HCAPLUS
Shaub, G	1995	8	318	J Supercrit Fluids	HCAPLUS
Shishikura, A	1994	42	1993	J Agric Food Chem	HCAPLUS
Shishikura, A	1992	5	303	Supercrit Fluids	HCAPLUS
Stejny, J	1998	39	4175	Polymer	
Subra, P	1996	12	217	Process Technology P	
Subramaniam, B	1997	86	885	J Pharm Sci	HCAPLUS
Tai, C	1998	44	989	AIChE J	HCAPLUS
Teipel, U	1997	22	165	Prop Expl Pyrotech	HCAPLUS
Thomasin, C	1998	87	259	J Pharm Sci	HCAPLUS
Thomasin, C	1998	87	269	J Pharm Sci	HCAPLUS
Tom, J	1992	514	238	ACS Symp Ser	
Tom, J	1991	7	403	Biotechnol Progress	HCAPLUS
Tom, J	1991	22	555	J Aerosol Sci	HCAPLUS
Tom, J	1992	7	9	J Supercrit Fluids	
Weidner, E	1996	12	217	Process Technology P	
Winters, M	1996	85	586	J Pharm Sci	HCAPLUS
Wubbolts, F	1997	667	242	ACS Symp Ser	HCAPLUS
Yeo, S	1993	41	341	Biotechnol Bioeng	HCAPLUS
Yeo, S	1994	83	1651	J Pharm Sci	HCAPLUS
Yeo, S	1993	26	6207	Macromol	HCAPLUS
Yeo, S	1995	28	1316	Macromol	HCAPLUS

L97 ANSWER 19 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1999:27755 HCAPLUS

DN 130:83612

TI Treatment of a substance with a dense fluid, especially with a **supercritical** fluid

IN King, Michael Blackshaw; Robertson, John

PA Smithkline Beecham PLC, UK; The University of Birmingham

SO PCT Int. Appl., 34 pp.

CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9858722	A1	19981230	WO 1998-GB1800	19980619

W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
 RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG

AU 9881182 A1 19990104 AU 1998-81182 19980619
 EP 991455 A1 20000412 EP 1998-930901 19980619

R: BE, CH, DE, ES, FR, GB, IT, LI, NL

JP 2002505617 T2 20020219 JP 1999-503993 19980619

PRAI GB 1997-12945 A 19970620

GB 1997-17344 A 19970816

WO 1998-GB1800 W 19980619

AB A process is disclosed for **precipitation** of a **solute** from a Dense Fluid **Solvent**. A solution of the **solute** in a Dense Fluid **Solvent** is **expanded** under conditions such that the Dense Fluid **Solvent** passes from the Dense Fluid **Solvent** region of its phase diagram into a 2-phase region of its phase diagram to cause **precipitation** of the **solute** from the solution Apparatus for performing the process is also disclosed.

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
British Nuclear Fuels P	1996			EP 0692289 A	HCAPLUS
Hewlett Packard Co	1990			EP 0384969 A	HCAPLUS
Jacques, L	1991			US 5011819 A	HCAPLUS
Moses, J	1988			US 4770780 A	HCAPLUS
Richard, S	1988			US 4734451 A	HCAPLUS

L97 ANSWER 20 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1998:686542 HCAPLUS

DN 129:262158

TI Fractional **crystallization** by **gas antisolvent** technique: theory and experiments

AU Bertucco, Alberto; Lora, Michele; Kikic, Ireneo

CS Istituto di Impianti Chimici, Universita di Padova, Padova PD, I-35131, Italy

SO AIChE Journal (1998), 44(10), 2149-2158

CODEN: AICEAC; ISSN: 0001-1541

PB American Institute of Chemical Engineers

DT Journal

LA English

AB The efficacy of **CO2** as an **antisolvent** was studied exptl. for the **precipitation** of naphthalene and phenanthrene from their solns. in toluene at 298 and 310 K. Phenanthrene was salted out of solution at every condition investigated, whereas naphthalene was never segregated as a solid phase. These behaviors are explained by a model representing the composition of the phases and supersatn. of the solution as functions of pressure. Based on results from ternary systems, expts. were performed with the quaternary system **CO2** -toluene-naphthalene-phenanthrene: starting from an equimolar solution of the two solids in toluene, phenanthrene with a purity higher than 98.5% can be collected in the **precipitation** cell, while naphthalene with .apprx.13% of phenanthrene is recovered from the liquid phase after **expansion**. The simulation of the process was able to account for the exptl. evidence. Although the **solutes** used do not have a practical application, a general method is outlined to exploit the possibility of using the **supercrit. antisolvent** technique for separation

IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)

(fractional crystallization by gas antisolvent technique)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Catchpole, O	1996			Proc Int Symp on Hig	
Chang, C	1994	72	56	Can J Chem Eng	HCAPLUS
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Foster, N	1997			Proc 4th Int Symp on	
Gallagher, P	1989			Supercritical Fluid	
Hong, S	1992	74	133	Fluid Phase Equil	HCAPLUS
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCAPLUS
Kikic, I	1997			Proc Int Symp on Sup	
Liang, M	1994			Proc Int Symp on Sup	
Liu, G	1996	35	4626	Ind Eng Chem Res	HCAPLUS
McHugh, M	1993			Supercritical Fluid	
Nagahama, K	1997			Proc Int Symp on Sup	
Shishikura, A	1994	42	1993	J Agric Food Chem	HCAPLUS
Shishikura, A	1992	5	303	J Supercrit Fluids	HCAPLUS
Shishikura, A	1991			Proc Int Symp on Sup	
Yeo, S	1993	41	341	Biotechnol and Bioen	HCAPLUS

L97 ANSWER 21 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1998:7381 HCAPLUS

DN 128:116698

TI **Supercritical crystallization: designed crystallization? Rapid expansion of supercritical solutions (RESS) and gas antisolvent (GAS) and principal applications**

AU Sanz Pastor, A. I.; Cocero, Alonso, M. J.

CS Dpto. Ingenieria Quimica, Universidad de Valladolid, Spain

SO Ingenieria Quimica (Madrid) (1997), 29(339), 183-190

CODEN: INQUDI; ISSN: 0210-2064

PB Ingenieria Quimica, S.A.

DT Journal; General Review

LA Spanish

AB The review, with 36 refs., covers methods of **supercrit. fluid crystallization** and discusses their possible uses in the pharmaceutical and polymer industries. **Supercrit. crystallization** methods can produce products with redefined particle sizes, narrow size distribution, absence of **solvent** occlusions, and residence times of seconds. In the RESS process (rapid **expansion** of **supercrit. solns.**), a **solute** dissolved in a **supercrit. fluid** **ppts.** to produce a sharp reduction in pressure and a following decline in solubility The **GAS (gas antisolvent)** process uses a pressurized **gas**, under critical or quasi-critical (pressure and temperature close to the critical point) conditions, **soluble** in organic **solvent** and insol. in the **solute**, such that dissoln. provokes a volumetric **expansion** which reduces the solubility of the **solute**; the **supercrit. fluid** acts as an **antisolvent**, causing **precipitation** of **solute**.

RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
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Berends, E	1994			PhD Thesis, Delft Un	
Brand, J	1988	166	139	Thin Solid Films	HCAPLUS
Brunner, G	1990	30	191	International Chemic	
Chang, C	1989	35	1.876	AIChE Journal	
Chang, C	1991	7	275	Biotechnology Progre	HCAPLUS
Cocero, M	1995	3	67	ALIMENTACION, TECNOL	
Cocero, M	1993		83	INGENIERIA QUIMICA	
Cocero, M	1995		169	INGENIERIA QUIMICA	HCAPLUS
de La Osa, M	1991		251	INGENIERIA QUIMICA	
Debenedetti, P	1993	24	27	Journal of Controlle	HCAPLUS
Desimone, J		265	356	Science	HCAPLUS
Dixon, D	1993	39	127	AIChE Journal	HCAPLUS
Eckert, C	1996	283	313	Nature	
Gallagher, P	1989		334	Supercritical Fluid	HCAPLUS
Kordikowski, A	1995	8	205	The Journal of Super	HCAPLUS
Krukonis, V	1989			Contract Rept	
Krukonis, V	1984			Paper 104f, AIChE me	
Larson, K	1986	2	73	Biotechnology Progre	HCAPLUS
Lele, A	1992	38	742	AIChE Journal	HCAPLUS
Lele, A	1994	33	1.476	Industrial Engineeri	
Lele, A	1990	31	677	Polym Prepr	HCAPLUS
Loth, M	1986	32	265	International Journa	
Makita, T	1989		222	Proceedings of the I	
Matson, D	1986	1	242	Adv Cer Mat	HCAPLUS
Matson, D	1987	21	109	Adv Ceram	HCAPLUS
Matson, D	1989		480	Chemtech	HCAPLUS
Medina, I	1993		443	Afinidad L	
Mohamed, R	1989	35	325	AIChE Journal	HCAPLUS
Mohamed, R	1989		355	Supercritical Fluid	HCAPLUS
Mueller, B	1989			DE 3744329	HCAPLUS
Mullin, J	1993			"Crystallization", T	
Ohgaki, K	1990	3	103	The Journal of Super	HCAPLUS
Schmitt, W	1995	41	2.476	AIChE Journal	
Tom, J	1991	7	403	Biotechnology Progre	HCAPLUS
Tom, J	1993		239	Supercritical Engine	
Yeo, S	1993	41	341	Biotechnology and Bi	HCAPLUS

L97 ANSWER 22 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1997:341799 HCAPLUS

DN 127:52671

TI Manufacture of microparticles by **crystallization** with highly compressed **gases**

AU Tschernjaew, Juri; Berger, Thomas; Weber, Andreas; Kummel, Rolf

CS Inst. Umwelt-, Sicherheits- Energietechnik e.V., Oberhausen, D-46047, Germany

SO Chemie-Ingenieur-Technik (1997), 69(5), 670-674

CODEN: CITEAH; ISSN: 0009-286X

PB VCH

DT Journal

LA German

AB Two techniques for **precipitation** of **solutes** by addition of highly compressed **gases**, the **GAS** (**gas antisolvent crystallization**) and the **PCA** (particles with a compressed fluid **antisolvent**) process were studied using the **precipitation** of ascorbic acid or L-asparagine from saturated solns. in EtOH by addition of CO₂. The **GAS** process gave particle sizes comparable to those of conventional **precipitation** and thermal **crystallization**, whereas the **PCA** process yielded particle sizes of the order of 1 µm and narrow size distribution. Disadvantages of the **GAS** process are the **crystallization** in the boundary layer combined with **precipitation** of polydisperse powders and the strong volume **expansion** of the liquid phase at the absorption of **gaseous**

antisolvents causing limited process capacity. The different mechanisms of **precipitation** depend on whether the **antisolvent** is a compressed, **supercrit.**, or liquefied **gas**.

→ L97 ANSWER 23 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1997:7897 HCAPLUS
 DN 126:62342
 TI Microparticle formation of HMX by **supercritical carbon dioxide antisolvent recrystallization**
 AU Cai, Jianguo; Sun, Zhaohui; Ma, Hongxi; Liao, Xiaochun; Zhou, Zhanyun
 CS Chem. Eng. Res. Center, ECU ST, Shanghai, 200237, Peop. Rep. China
 SO Huadong Ligong Daxue Xuebao (1996), 22(5), 512-517
 CODEN: HLI XEV
 PB Huadong Ligong Daxue Xuebao Bianjibu
 DT Journal
 LA Chinese
 AB The **recrystn.** ratio of 1, 3, 5, 7-tetranitro-1, 3, 5, 7-tetraazacyclooctane (HMX) in acetone, cyclohexanone, and dimethylsulfoxide solution using **supercrit. carbon dioxide antisolvent (GAS)** was compared. By using **GAS** process in acetone solution, microparticles of β -HMX within 2 .apprx. 13 μ m can be obtained. Effects of pressure, temperature, initial feed concentration of HMX **solute**, **expansion** speed of solution and growth of **crystal** on the **GAS** process have been studied. Under all exptl. pressures of 8.0 .apprx. 12.0 MPa tested, lower test temperature and lower concentration of feed solution were preferable for obtaining β -HMX and microparticles.
 IT 124-38-9, **Carbon dioxide**, uses
 RL: NUU (Other use, unclassified); TEM (Technical or engineered material use); USES (Uses)
 (microparticle formation of HMX by **supercrit. carbon dioxide antisolvent recrystn.**)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 24 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1996:711759 HCAPLUS
 DN 125:332821
 TI Fine particle **coating** in a circulating fluidized bed by rapid **expansion** of **supercritical** fluid solutions
 AU Tsutsumi, Atsushi; Nakata, Mitsutoshi; Mineo, Tomoko; Yoshida, Kunio
 CS Dep. Chem. System Engineering, Univ. Tokyo, Tokyo, 113, Japan
 SO Kagaku Kogaku Ronbunshu (1996), 22(6), 1379-1383
 CODEN: KKRBAW; ISSN: 0386-216X
 PB Kagaku Kogaku Kyokai
 DT Journal
 LA Japanese
 AB Fine particle **coating** by rapid **expansion** of **supercrit.** CO₂ solns. of paraffins was performed in a circulating fluidized bed (50 mm i.d.) with an internal nozzle at the center of the riser. Microspheroidal catalyst particles (average particle size 56 μ m) were used as the core particles. The **coating** mass and **coating** rates were measured by a sampling method. The effects of **gas** flow rate and **solute** concentration on **coating** rate and **coating** efficiency were examined
 IT 124-38-9, **Carbon dioxide**, processes
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical

process); PROC (Process); USES (Uses)
(**supercrit.**, **solvent**; in **coating** of fine
particles in circulating fluidized beds by rapid **expansion** of
supercrit. solns.)

RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 25 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1996:616340 HCAPLUS
DN 125:277764
TI Studying Activity Coefficients of Probe **Solutes** in Selected
Liquid Polymer **Coatings** Using Solid Phase Microextraction
AU Zhang, Zhouyao; Pawliszyn, Janusz
CS Department of Chemistry, University of Waterloo, Waterloo, ON, N2L 3G1,
Can.
SO Journal of Physical Chemistry (1996), 100(44), 17648-17654
CODEN: JPCHAX; ISSN: 0022-3654
PB American Chemical Society
DT Journal
LA English
AB The study of **solute**-polymeric liquid **solvent** interaction
contributes to the understanding of the fundamental principles of
chromatog. since liquid polymers are often used as stationary phases in
gas chromatog. (GC) and high-performance liquid chromatog. (HPLC).
The knowledge of how a polymeric stationary phase interacts with different
types of compds. helps researchers to select and synthesize the right
phase for successful separation of mixts. in a time-efficient manner. The
development of a simple, cost effective, and time-efficient method for
studying **solute-solvent** interaction can aid
significantly the ever-**expanding** applications of chromatog. In
this work, a new approach, solid phase microextn. (SPME), is used for
investigations of activity coeffs. of the McReynolds probe **solutes**
in selected liquid polymers. The probe **solutes** are absorbed by an
immobilized liquid polymer phase **coated** on the outside surface of
a fused silica fiber, and quantitated by a GC technique using a com.
available GC column. The research in this study shows that activity
coeffs. measured by SPME are equivalent to those by the commonly used GC
method. This new method eliminates the need to prepare a GC column using
the polymer of interest as in the GC method and, thus, significantly
simplifies the whole measuring process. It also allows convenient
investigation of the prepared **coating** by other surface and
spectroscopic techniques.

L97 ANSWER 26 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1996:122671 HCAPLUS
DN 124:274772
TI **Crystallization** of phenanthrene from toluene with **carbon**
dioxide by the **GAS** process
AU Berends, Edwin M.; Bruinsma, Odolf S. L.; de Graauw, Jan; van Rosmalen,
Gerda M.
CS Lab. Process Equipment, Delft Univ. Technol., Delft, 2628 CA, Neth.
→ SO AIChE Journal (1996), 42(2), 431-9
CODEN: AICEAC; ISSN: 0001-1541
PB American Institute of Chemical Engineers
DT Journal
LA English
AB The **crystallization** of phenanthrene from toluene with CO2 as
the **antisolvent gas** is described. In the **GAS**

process, a pressurized **gas** is dissolved into a liquid **solvent**, where it causes a volumetric **expansion** and lowers the solubility of the **solute**. Theor. models are presented for the liquid-phase **expansion** and the solubility as a function of pressure and temperature. The Nyvlt theory for batch **crystallization** is adapted to predict the pressure profile in the **crystallizer** needed to maintain a constant supersatn. and growth rate. Generation of seeds is accomplished via a pressure pulse at the saturation pressure. The average particle

size of the phenanthrene could be varied from 160 to 540 μm . Creation of seeds doubles the particle size and reduces the coefficient of variation significantly. The residual amount of toluene in the **crystals** without treatment is .apprx.70 ppm. The particles are agglomerates of phenanthrene **crystals**.

IT 124-38-9, Carbon dioxide, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (crystallization of phenanthrene from toluene by **gas**
 antisolvent process using)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 27 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1996:4915 HCAPLUS
 DN 124:156296
 TI An exact lattice model of complex solutions: chemical potentials depend on **solute** and **solvent** shape
 AU Krukowski, Anton E.; Chan, Hue Sun; Dill, Ken A.
 CS Dep. Pharmaceutical Chem., Univ. California San Francisco, San Francisco, CA, 94143-1204, USA
 SO Journal of Chemical Physics (1995), 103(24), 10675-88
 CODEN: JCPSA6; ISSN: 0021-9606
 PB American Institute of Physics
 DT Journal
 LA English
 AB For the theor. modeling of phys. transformations such as boiling, freezing, glassification, or mixing, it is necessary to know how the partition function of a system depends on its d. Many current treatments rely either on low d. **expansions** or they apply to very simple and sym. mol. shapes, like spheres or rods. Here the authors develop an exact anal. lattice theory of materials and mixts. that applies to arbitrarily complex mol. shapes over the full range of densities from **gas** to **crystal**. The approach is to compute partition functions for small nos. of shapes and to explore the dependence on d. by varying the volume of the system. Recently a question has been raised about whether entropies of dissoln. are better treated using classical solvation theories or Flory-Huggins theory. The authors explore this for a range of mol. sizes and shapes, from lattice squares and cubes, to rods, polymers, crosses, and other shapes. Beyond low densities, the entropic component of the chemical potential depends on shape due to the different degrees to which mols. "interfere" with each other. It was found that neither Flory-Huggins nor classical solvation theories is correct for all shapes. Mols. that are "articulated" are remarkably well treated by Flory-Huggins theory, over all densities, but globular mols. are qual. and quant. different, and are better treated by the classical chemical potential, consistent with expts. of Shinoda and Hildebrand. These results confirm that the Flory-Huggins theory differs from classical theory not because of mol. size differences per se; it accounts for the coupling between translations and conformational steric interference.

- L97 ANSWER 28 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1995:26711 HCAPLUS
DN 122:107766
TI **Solute** deposition in a porous polymer matrix from rapid **expansion** of a **supercritical** solution
AU Bertuccio, A.; Guarise, G. B.; Pallado, P.; Corain, B.
CS Istituto di Impianti Chimici, Universita di Padova, Padua, 35131, Italy
SO Chemical and Biochemical Engineering Quarterly (1994), 8(1), 11-16
CODEN: CBEQEZ; ISSN: 0352-9568
DT Journal
LA English
AB The rapid **expansion** of a **supercrit.** solution in a porous polymer matrix is carried out to obtain the deposition of the **solute** inside the structure. The sudden pressure reduction results in a strong supersatn., so that the formation of small solid particles can be achieved. The deposition of ferrocene **crystallites** on poly(N,N-dimethylacrylamide) is studied using CO₂ at temps. between 323-353 K and pressures from 18 to 22 MPa. A math. model is developed to represent the **expansion** of a real **gas** through the exit nozzle. Simulated and exptl. profiles for pressure and temperature are in agreement, so that the amount of **precipitated solute** and the phys. state of the **solvent** can be predicted.
- L97 ANSWER 29 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1994:587124 HCAPLUS
DN 121:187124
TI **Precipitation** of poly(L-lactic acid) and composite poly(L-lactic acid)-pyrene particles by rapid **expansion** of **supercritical** solutions
AU Tom, Jean W.; Debenedetti, Pablo G.; Jerome, Robert
CS Dep. Chem. Eng., Princeton Univ., Princeton, NJ, 08544, USA
SO Journal of Supercritical Fluids (1994), 7(1), 9-29
CODEN: JSFLEH; ISSN: 0896-8446
DT Journal
LA English
AB The rapid **expansion** of **supercrit.** solns. (RESS) was explored as a novel route to the formation of microparticles and microspheres useful in controlled drug delivery applications. Poly(L-lactic acid) was dissolved in **supercrit.** CO₂ with CHClF₂ as a **cosolvent** and **precipitated** by RESS. The polymers solubility and its mol. weight in solution were found to depend on processing time because of sample polydispersity. The morphol. of the **precipitate** (microparticles, microspheres, agglomerates, or dendrites) was examined as a function of the type of the **expansion** device (orifices or capillaries), pre-**expansion** temperature, and **solvent** composition. Dendrites were the most common morphol. when using orifices. Microspheres formation using capillaries occurred with low pre-**expansion** temps. and low length-to-diameter ratio. A one-dimensional fluid mech. model of the **solvent's expansion** in a capillary indicates that microspheres were formed preferentially when the fluid's exit d. was high, suggesting that substantial **precipitation** occurred outside the capillary. In the first comprehensive study of the effects of process conditions on the composite powders formed by RESS copptn., pyrene (a nonpolymeric fluorescent **solute**) was copptd. with poly(L-lactic acid) from **supercrit.** CO₂-CHClF₂ solns. Fluorescence and transmission microscopy allowed the observation of pyrene in the coppt. These expts. showed clearly the uniform incorporation of pyrene microparticles within polymer microspheres, and thus, the feasibility of RESS as a technique for the copptn. of composite particles with multiple substances.
IT 124-38-9, Carbon dioxide, properties
RL: PRP (Properties)

(solvent; composite particles for controlled drug release
copptn. by rapid expansion of supercrit. solns.)

RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

→ L97 ANSWER 30 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1994:194464 HCAPLUS
DN 120:194464
TI Relative supersaturation ratio and separation factor in
crystallization with high pressure CO2
AU Chang, Chiehming J.; Liou, Yuchung; Lan, Wen Jen
CS Dep. Chem. Eng., Natl. Chung-Hsing Univ., Taichung, 400, Taiwan
SO Canadian Journal of Chemical Engineering (1994), 72(1), 56-63
CODEN: CJCEA7; ISSN: 0008-4034
DT Journal
LA English
AB **Crystallization** in the presence of high-pressure gas as
antisolvent could be applied for the recovery of valuable compds.
from liquid solution A study of separation behavior is presented here for a
mixture
of anthracene and anthraquinone in cyclohexanone **expanded** with a
gaseous antisolvent, CO2. The pressure range
was 0.1-12 MPa; the temperature was either 292 or 313 K. Separation factors
were
obtained from the measured salted-out yields and the supersatn. of each
solute could be also obtained for this pressure-tuning
crystallization The separation factor varied almost linearly with relative
supersatn. ratio in the **crystallization** of anthracene-anthraquinone from
cyclohexanone and CO2.
IT 124-38-9, Carbon dioxide, uses
RL: USES (Uses)
(in pressure-induced **crystallization** of anthracene and anthraquinone
from cyclohexanone)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 31 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1994:94354 HCAPLUS
DN 120:94354
TI Sample introduction in capillary **supercritical** fluid
chromatography using sequential density gradient focusing and
solvent venting
AU Liu, Zaiyou; Farnsworth, Paul B.; Lee, Milton L.
CS Dep. Chem., Brigham Young Univ., Provo, UT, 84602, USA
SO Journal of Microcolumn Separations (1991), 3(5), 435-42
CODEN: JMSEBJ; ISSN: 1040-7685
DT Journal
LA English
AB A technique was developed for large volume sample introduction in capillary
supercrit. fluid chromatog. A 20-cm length of 200-µm i.d.
capillary tubing was used as precolumn. The precolumn temperature could be
easily controlled by passing an elec. current through an elec. conductive
paint **coated** on its outer surface. During injection, the same

solvent was vented from the precolumn with **CO2** (**gas**) at 32 atm, while the precolumn was kept at room temperature. **Solutes** were transferred onto the head of the anal. column as a narrow band by d. gradient focusing, which was established with (a) a temperature gradient along the precolumn, (b) a rapid **expansion** of **CO2** from **supercrit.** fluid to **gas**, and (c) a temperature difference between the precolumn and the anal. column. This injection approach minimized **solute** mass discrimination and could be easily performed.

→ L97 ANSWER 32 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1994:33310 HCAPLUS
 DN 120:33310
 TI Purification of polycyclic aromatic compounds using salting-out separation in high-pressure **carbon dioxide**
 AU Chang, Chiehming J.; Liou, Yuchung
 CS Dep. Chem. Eng., Yuan Ze Inst. Technol., Taoyuan, 320, Taiwan
 SO Journal of Chemical Engineering of Japan (1993), 26(5), 517-22
 CODEN: JCEJQA; ISSN: 0021-9592
 DT Journal
 LA English
 AB **Gas antisolvent crystallization** has the potential for application in the recovery of valuable compds. from solution, and in the separation of solid-solid mixts. Exptl. data are presented for a mixture of anthracene and anthraquinone dissolved in cyclohexanone which was **expanded** by a **gaseous antisolvent, CO2**. The pressure range is 0.1-12 MPa, and the temperature 291-313 K. The relation of salted-out yield and normalized feed concentration gives an important parameter, the min. solubility, from which supersatn. can be defined for **gas antisolvent crystallization**. Effects of initial feed concns. of solid **solutes**, temperature, and pressure on the separation of anthracene and anthraquinone have also been studied.
 IT 124-38-9, **Carbon dioxide**, uses
 RL: USES (Uses)
 (high-pressure, **crystallization** of polycyclic aromatic compds. using)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 33 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1993:452405 HCAPLUS
 DN 119:52405
 TI Manufacture of **coated** fine particles, especially, lanthanum oxide-**coated** silica particles
 IN Kitagawa, Kazuo; Yamamoto, Seiichi; Moritoki, Masato
 PA Kobe Steel Ltd, Japan
 SO Jpn. Kokai Tokkyo Koho, 8 pp.
 CODEN: JKXXAF
 DT Patent
 LA Japanese
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 05057166	A2	19930309	JP 1991-246861	19910831
PRAI	JP 1991-246861		19910831		

AB The process comprises dissoln. of a 1st **solute** (e.g., SiO2) and 2nd **solute** (e.g., La2O3) in 1st and 2nd **solvents** (e.g., both water) to form 1st and 2nd systems at **supercrit.** or

subcrit. states, adiabatic **expansion** of the 1st system to form a 1st **solute** fine particles via supersatd. state, increasing the pressure to that of the 2nd system and mixing with the latter, then adiabatic **expansion** of the mixed system for **precipitating** and **coating** of the 2nd **solute** on the surfaces of the 1st **solute** fine particles via supersatd. state. The **coated** fine particles can be further **coated** with nth ($n \geq 3$) **solutes** from nth solns. by the same operation.

L97 ANSWER 34 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1993:415024 HCAPLUS
 DN 119:15024
 TI Three-phase separation process for solutions, especially seawater and waste liquids
 IN Wilensky, Joseph
 PA USA
 SO U.S., 28 pp. Cont.-in-part of U.S. 5,084,187.
 CODEN: USXXAM
 DT Patent
 LA English
 FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 5167838	A	19921201	US 1991-814564	19911230
	US 5084187	A	19920128	US 1991-701452	19910515
PRAI	US 1991-701452		19910515		

AB Seawater, brines, industrial wastewaters, and nonaq. industrial water liqs. are separated into potable water, concentrated brine, and purified **solutes** by dissolving a fluid, e.g., liquid or **gaseous CO₂**, into the solution to produce a single-phase liquid, lowering the liquid temperature, and then performing a Joule-Thompson free **expansion** on the liquid. As a result, the liquid is separated into a evaporated **gas** phase mainly comprised of the **solute**, and a **crystallized** solid phase mainly comprised of the **solvent** (e.g., ice). Any remaining liquid is recycled. The ice can be melted and used in production of carbonated beverages. When the remaining liquid is a brine, MgCO₃ can be recovered from it.

IT **124-38-9, Carbon dioxide**, occurrence
 RL: OCCU (Occurrence)
 (in seawater desalination and waste liqs. separation)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 35 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1993:220656 HCAPLUS
 DN 118:220656
 TI Mysterious fine particles formed during the rapid **expansion** of **supercritical** water solutions
 AU Tanaka, Yoshiyuki
 CS Fac. Eng., Kobe Univ., Kobe, 657, Japan
 SO Koatsuryoku no Kagaku to Gijutsu (1992), 1(4), 263-71
 CODEN: KKGIE2; ISSN: 0917-639X
 DT Journal
 LA Japanese
 AB Fine silica particles were produced by the rapid **expansion** of **supercrit.** water-SiO₂ solns. (RESS) at 723-823 K and pressures from 50 to 100 MPa. New spherical particles sprouting whiskers were also discovered in the autoclave after the RESS. The solubility of solids in

supercrit. fluids is a very sensitive function of temperature and pressure. Small changes of pressure result in large changes in d. and **solvent** power, because **supercrit.** fluids are highly compressible. The rapid **expansion** of **supercrit.** solns. can give rise to very large supersatn. ratios. Nucleation rates are determined by the competition among **solvent expansion**, cooling due to depressurization, and high supersatn. In order to control the product, morphol., the effects of exptl. parameters, such as preexpansion temperature and pressure, **solute** concentration, depressurization schemes, nozzle configuration, and sampling method on the product characteristics of materials, were investigated by means of SEM and x-ray diffraction anal. Control of particle size distribution is possible by regulating supersatn. ratio as well as suitable selection of preexpansion temperature and pressure. Unique features of the RESS process are discussed.

L97 ANSWER 36 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1992:86953 HCAPLUS

DN 116:86953

TI Manufacture of fine particles.

IN Moritoki, Masato; Kitagawa, Kazuo; Inoe, Yasuhiko

PA Kobe Steel, Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 9 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 03271113	A2	19911203	JP 1990-67890	19900317
PRAI	JP 1990-67890		19900317		

AB Fine particles are manufactured by dissoln. of a **solute** in a **solvent** of **supercrit.** or subcrit. state, adiabatic **expansion** in a closed high-pressure container, **precipitation** of the **solute** in the container, releasing of residual pressure from the container to atmospheric, then (or meanwhile) recovery of fine particles of the **solute**. Number and shape of the fine particles are controlled by controlling speed of the adiabatic **expansion**. Thus, SiO₂ fine particles was manufactured from aqueous solution of **supercrit.** state.

L97 ANSWER 37 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1990:574645 HCAPLUS

DN 113:174645

TI Homogeneous nucleation in **supercritical** fluids

AU Debenedetti, Pablo G.

CS Dep. Chem. Eng., Princeton Univ., Princeton, NJ, 08544, USA

SO AIChE Journal (1990), 36(9), 1289-98

CODEN: AICEAC; ISSN: 0001-1541

DT Journal

LA English

AB When a **supercrit.** solution is rapidly **expanded**, large **solute** supersatns. can be attained, and small particles are formed. The evolution of the homogeneous nucleation rate, work of nucleus formation, and critical nucleus size along different **expansion** paths is studied for the model system phenanthrene-CO₂. Nucleation rates are the result of the competition among **solvent expansion**, cooling due to depressurization, and high supersatn. Although supersatns. can reach very high values (>106), relatively flat nucleation rate profiles result due to cooling and **expansion**. For an interfacial tension of 0.02 N/m, computed nucleation rates never exceed 104/s·cm³. A substantial fraction of the maximum nucleation rate is attained with partial decompression to >1 bar.

L97 ANSWER 38 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1990:462140 HCAPLUS
 DN 113:62140
 TI **Solvent expansion** and **solute** solubility
 predictions in **gas-expanded** liquids
 AU Chang, Chiehming J.; Randolph, Alan D.
 CS Dep. Chem. Eng., Univ. Arizona, Tucson, AZ, 85721, USA
 SO AIChE Journal (1990), 36(6), 939-42
 CODEN: AICEAC; ISSN: 0001-1541
 DT Journal
 LA English
 AB The **expansion** of binary systems (e.g., PhMe-CO₂ and
 BuOH-CO₂) in the miscible liquid-phase region and solubility of the
solute (e.g., β -carotene in PhMe and acetaminophen in BuOH)
 in the liquid phase are studied. **Solvent expansion** at
 298 K, solid solubility in the **gas antisolvent** addition for
 liquid-phase **precipitation** of solids, partial molar volume changes in the
gas antisolvent addition process, and **crystallization**
 kinetics in the **gas antisolvent** addition **recrystn**
 . are presented graphically and discussed.
 IT 124-38-9, **Carbon dioxide**, properties
 RL: PRP (Properties)
 (expansion of, determination of, in **gas-expanded**
 liqs.)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

→ L97 ANSWER 39 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1989:635821 HCAPLUS
 DN 111:235821
 TI **Precipitation** of microsize organic particles from
supercritical fluids
 AU Chang, C. J.; Randolph, A. D.
 CS Dep. Chem. Eng., Univ. Arizona, Tucson, AZ, 85721, USA
 SO AIChE Journal (1989), 35(11), 1876-82
 CODEN: AICEAC; ISSN: 0001-1541
 DT Journal
 LA English
 AB The **precipitation** of organic particles from **supercrit.** fluids
 (SF) by **expansion** (SFX) has become an interesting alternative to
 milling without thermal decomposition The rapid **expansion** produces a
 dramatic change of the **solute** supersatn. ratio that results in
precipitation with a narrow particle-size distribution. It was found that
 β -carotene **ppts.** from SF ethylene and ethane have the feed
 material **crystallinity**. However, SF CO₂ reacted with
 β -carotene and did not give characteristic β -carotene x-ray
 spectra. The mean particle sizes of these **ppts.** were in the
 submicron range (.apprx.0.3 μ m). Increased solubility was obtained by
 addition
 of PhMe as **cosolvent** in SF ethylene. The mean size of
 β -carotene particles remained unchanged if the PhMe concentration was <1.5
 mol%. The SFX process appears to be in a single fluid phase when <1.5
 mol% PhMe **cosolvent** is used.

L97 ANSWER 40 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1988:552362 HCAPLUS
 DN 109:152362
 TI **Supercritical** fluid molecular spray thin films and fine powders
 IN Smith, Richard D.

PA Battelle Memorial Institute, USA
 SO U.S., 25 pp. Cont.-in-part of U.S. 4,582,731.
 CODEN: USXXAM
 DT Patent
 LA English
 FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 4734451	A	19880329	US 1986-839079	19860312
	US 4582731	A	19860415	US 1983-528723	19830901
	CA 1327684	A1	19940315	CA 1988-556177	19880108
PRAI	US 1983-528723		19830901		
	US 1986-839079		19860312		

AB Solid films are deposited on surfaces or fine powders are formed by **supercrit.** fluid mol. spray in which a solution of the **supercrit.** fluid and the solid material as **solute** is formed, the solution is rapidly **expanded** through an orifice to produce a particulate spray and vaporized **solvent**, and the mol. spray is directed against a surface to deposit a film or it is discharged into a low pressure region to form a powder. The temperature of the **supercrit.** solution is selected and maintained for formation of the 2-phase system during **expansion** to control the porosity of the film or powder. Examples are discussed for the deposition of polystyrene films on Pt and fused silica, for the deposition of silica on Pt and glass, and for production of GeO₂ powders.

L97 ANSWER 41 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1987:461383 HCAPLUS

DN 107:61383

TI Production of powders and films by the rapid **expansion** of **supercritical** solutions

AU Matson, Dean W.; Petersen, Robert C.; Smith, Richard D.

CS Chem. Sci. Dep., Battelle, Pac. Northwest Lab., Richland, WA, USA

SO Journal of Materials Science (1987), 22(6), 1919-28

CODEN: JMTSAS; ISSN: 0022-2461

DT Journal

LA English

AB A process utilizing the rapid **expansion** of **supercrit.** fluid solns. (RESS) is described for the manufacture of fine powders and thin films by the rapid nonequil. **precipitation** of nonvolatile compds. from dense **gas** solns. upon **expansion**. A variety of the fluid solution **expansion** parameters, including **solute** and **solvent** identity, **solute** concentration, **expansion** temperature, and **expansion** nozzle configuration, affect the product characteristics of materials formed during the RESS process. Conditions favoring thin film formation include very dilute solns. and short nozzles minimizing residence time during **expansion**. Particle formation is favored by more concentrated solns. The process produced products of widely varying morphol. by the adjustment of RESS parameters, and examples of SiO₂, GeO₂, and various polymeric materials are presented. Unique features of the RESS process relevant to other powder and film production methods are described and potential applications are discussed.

L97 ANSWER 42 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1986:227104 HCAPLUS

DN 104:227104

TI **Supercritical** fluid molecular spray film deposition and powder formation

IN Smith, Richard D.

PA Battelle Memorial Institute, USA

SO U.S., 15 pp.

CODEN: USXXAM

DT Patent

LA English

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 4582731	A	19860415	US 1983-528723	19830901
	JP 61500210	T2	19860206	JP 1984-503580	19840828
	JP 04019910	B4	19920331		
	CA 1260381	A1	19890926	CA 1984-461977	19840828
	US 4734451	A	19880329	US 1986-839079	19860312
PRAI	US 1983-528723		19830901		
	WO 1984-US1386		19840828		

AB Thin films are deposited, or fine powders are formed, by dissolving a solid material into a **supercrit.** fluid at an elevated pressure and then rapidly **expanding** the solution through a short orifice into a region of relatively low pressure. This produces a mol. spray which is directed against a substrate to deposit a solid thin film on it, or discharged into a collection chamber to collect a fine powder. Upon **expansion** and supersonic interaction with background **gases** in the low pressure region, the clusters of **solvent** are broken up and the **solvent** is vaporized and pumped away. **Solute** concentration in the solution is varied primarily by varying solution pressure. **Solvent** clustering and **solute** nucleation are controlled by manipulating the rate of **expansion** of the solution and the pressure of the lower pressure region.

L97 ANSWER 43 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1980:189439 HCAPLUS

DN 92:189439

TI The effect of quasispherical and chainlike **solutes** on the nematic to isotropic phase transition in liquid **crystals**

AU Oweimreen, G. A.; Martire, D. E.

CS Dep. Chem., Georgetown Univ., Washington, DC, 20057, USA

SO Journal of Chemical Physics (1980), 72(4), 2500-10

CODEN: JCPSA6; ISSN: 0021-9606

DT Journal

LA English

AB The effects of **solute** mol. structure and **solvent** mol. structure on nematic phase stability in dilute binary mixts. of nonmesomorphic **solutes** and nematogenic **solvents** were studied. Addition of the perturbing **solute** to the liquid-**crystal solvent** leads to depression of the nematic-isotropic (NI) transition temperature and formation of a two-phase region. Directly determined moduli of the slopes, β_n vs. **solute** mol fraction (x_2) diagrams are reported for quasispherical and chainlike **solutes** with two nematogenic **solvents**. The systems studied were the quasispheres Et4C (tetraethylmethane) and R4Sn (R = CH3, C2H5, C3H7 and C4H9) and the chains (n-C8H18 through n-C14H30, mixed with MBBA and p-n-pentyl-p'-cyanobiphenyl (5CB)). Also reported are indirectly determined $\beta_{n\infty}$ and $\beta_{i\infty}$ values (limit as $x_2 \rightarrow 0$), using a novel approach combining differential scanning calorimetry (for the pure **solvent** contribution) and **gas**-liquid chromatog. (for the solution contribution), for Et4C and n-C5H12 through n-C11H24, with MBBA, 5CB, p-azoxyanisole, and p,p'-di-hexyloxyazoxybenzene. For the systems in common, the average difference between the directly and indirectly determined β values is .apprx.10%, the comparison suggests slight curvature of the phase boundary lines. The exptl. β values, as a function of increasing **solute** size, double (roughly) for the quasispheres and increase only slightly for the chains, reflecting the concurrent behavior of the solution contribution to β . The thermodyn. results for the quasispherical **solutes** are compared with predicted values from statistical-mech. theories based on rigid-rod **solvent** mols.: (1) lattice model, (2) virial **expansion** treatment, (3) mol.-field model (after Maier and Saupe), and (4) van der

Waals model. All four models correctly predict the observed trend of increasing β_n and β_i with increasing **solute** size and yield predicted slopes which are within a factor of 2 of experiment. All are deficient to a minor or major extent in their predictions of the **solvent** and solution contributions to the β values. The more tractable lattice model is used to examine the chainlike **solutes** and the effect of **solvent** end-chain flexibility. It correctly predicts the qual. features of the observed dependence of β on **solute** size for the different **solute** structures (including rigid-rod **solutes**) and indicates that dissolved n-alkane **solutes** have appreciable (effective) chain flexibility in nematic **solvents**. Incorporation of some **solvent** end-chain flexibility in the lattice model markedly improves agreement with experiment, primarily through better quant. prediction of the solution contribution.

L97 ANSWER 44 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1920:7933 HCAPLUS

DN 14:7933

OREF 14:1468d-i,1469a-c

TI Some new hypotheses as to different states of matter

AU Bacon, N. T.

CS Peace Dale, RI

SO Journal of Physical Chemistry (1919), 23, 469-77

CODEN: JPCCHX; ISSN: 0022-3654

DT Journal

LA Unavailable

AB B. is not "satisfied with the current reason given to explain why mols. condense from a vessel filled with the saturated vapor when the temperature is reduced, for this same reduction should cause a reduction of pressure, even without condensation." After a consideration of the properties of a liquid and its vapor as they approach and pass the critical point, he asks, "is it not a fair inference, in view of these things, that in the condition of a true **gas** the spheres of influence of mols. decrease with advancing temperature so as to allow a free path and thus cause them to follow Boyle's law?". If in the **gaseous** condition the diameter of the sphere of influence of the mol. is thus an inverse function of the temperature, "we should find a probability that in the vapor condition

at

temps. below the critical the mol. would continue to **expand**. If this is true we should have a direct explanation of the separation of condensate whenever a saturated vapor is cooled under constant volume. There

would

no longer be room in their **gaseous** state for all the **expanding** mols. so that some of them would be obliged to go into the less bulky liquid form." A further development of this conception has grown out of the consideration of the very small solubility of BaSO_4 . The question is raised, "how (according to Calvert's determination) one single ion

of

Ba , in the presence of a corresponding ion of SO_4 , can affect simultaneously 10,000,000 mols. of H_2O as to deprive every one of them of the power to dissolve any more BaSO_4 ?" Regarding this problem he says, "I find the easiest explanation in assuming a virtual **expansion** of the mols. of the **solute** so as practically to occupy all the inter-mol. space of the **solvent** in much the same way in which I have supposed mol. in the volatile conditions to increase the diameter of the spheres of influence of their mols. as the temperature falls." Later he

states,

"that by way of explanation I find myself reduced to the conception of the BaSO_4 breaking up into an enormous number of electrons, or emanations of which electrons are built, each having the characteristic periodicity of BaSO_4 (and not solely of any constituent thereof) and that these so permeate the **solvent** that each mol. of this is in some way in

contact, periodic at least, with such particles, so as to maintain an equilibrium relation." "Colloidal solns. are merely individual mols. held in suspension and carry a current only mechanically through a menstruum. which does not dissolve them. They take a charge by metallic conduction and thus are repelled from one pole and attracted to the other."

"Hydrolysis represents a condition where the complicated periodicity of the salt becomes too extended, so that part of the **solute** loses coherence and the fractions revert to their simpler (though related) periodicity, each in its own condition, as if the other were not present. These conditions are quite different from those of electrolysis. In this the ions exist as atoms combined with charges of electricity (instead of complementary atoms) to make virtual mols. suspended in the menstruum much as are the metallic mols. in colloidal solns. and very different from the clouds of diffused electrons or emanations filling intermol. spaces which by my theory make a continuity of particles of the **solute** roughly answering to that of Bragg for matter in **crystalline** form. This involves recognizing inherent differences between **solvent** and **solute**. In many cases substances are mutually soluble, so that each acts both (or either) as **solvent** and (or) **solute**; in other cases one has a distinctly different type of action from the other." Regarding the more rapid **expansion** of a liquid as it approaches the critical point, there is suggested "the possibility that the rapid increase in volume may be due to mols. in the vapor state dissolved as vapor by other mols. of the same kind in the liquid state. This is analogous to Richards' explanation of the action of water between 0° and 4° as due to solution of ice mols. as such and, like S_{μ} in $S\lambda$."

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MOST RECENT DERWENT UPDATE: 200416 <200416/DW>

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L116 ANSWER 1 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 2004-068695 [07] WPIX

CR 2002-099223 [14]; 2002-547217 [58]; 2002-547218 [58]; 2002-664436 [71];

2003-267551 [26]; 2003-447308 [42]

DNN N2004-055254 DNC C2004-028225

TI Collecting samples comprises controlling e.g. pressure of stream to improve separation of monophasic fluid into **gaseous** and liquid phases, **expanding** stream by directing through **expansion** space, and retaining liquid in collection device.

DC B04 J04 S03

IN BENTE, P F; BERGER, T A; FOGELMAN, K D; KLEIN, K; NICKERSON, M; STAATS, L T

PA (BERG-N) BERGER INSTR INC; (BENT-I) BENTE P F; (BERG-I) BERGER T A; (FOGE-I) FOGELMAN K D; (KLEI-I) KLEIN K; (NICK-I) NICKERSON M; (STAA-I) STAATS L T

CYC 32

PI US 2002139752 A1 20021003 (200407)* 29p B01D011-00 <--
 EP 1348956 A2 20031001 (200407) EN G01N030-06
 R: AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LI LT LU LV
 MC MK NL PT RO SE SI SK TR
 US 6632353 B2 20031014 (200407) B01D015-08 <--

ADT US 2002139752 A1 CIP of US 2000-607316 20000626, US 2002-113599 20020329;
 EP 1348956 A2 EP 2003-4475 20030227; US 6632353 B2 CIP of US 2000-607316 20000626, US 2002-113599 20020329

FDT US 2002139752 A1 CIP of US 6413428; US 6632353 B2 CIP of US 6413428

PRAI US 2002-113599 20020329; US 2000-607316 20000626

IC ICM **B01D011-00**; **B01D015-08**; G01N030-06
 ICS G01N030-16; G01N030-28; G01N030-80

AB US2002139752 A UPAB: 20040128
 NOVELTY - Collecting samples from flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid comprising controlling the pressure, temperature and velocity of the flow stream to improve separation of a monophasic fluid mixture into separate **gaseous** and liquid phases and **expanding** by directing through an **expansion** space, is new.
 DETAILED DESCRIPTION - Collecting samples from a flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid comprising controlling the pressure, temperature and velocity of the flow stream to improve separation of a monophasic fluid mixture into separate **gaseous** and liquid phases, **expanding** the flow stream by directing it through an **expansion** space, and retaining the liquid phase in a collection device, is new.
 INDEPENDENT CLAIMS are also included for:
 (1) collecting samples comprising injecting the samples into a flow stream, controlling the pressure, temperature and velocity of the flow stream to improve separation of a monophasic fluid mixture into separate **gaseous** and liquid phases, **expanding** the flow stream by directing it through an **expansion** space in a flow line carrying the flow stream, and retaining the liquid phase in a collection device;
 (2) a system for collecting samples from a flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid, comprising a flow line creating a space in which the flow stream moving through the line is **expanded** and the linear velocity of the flow stream is slowed, and a collection device downstream of the space in the flow line, in which the liquid phase from the flow line is retained; and
 (3) a further system for collecting samples from a flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid, comprising an injection valve for injecting discrete samples into the flow stream, a separation device to elute **solutes** of the samples, a detector to detect the concentrations of the **solutes** in the flow stream, a phase separation stage to control the pressure, temperature and velocity of the flow stream to improve separation, comprising a series of heaters and transfer lines to separate a monophasic flow stream into liquid and **gas** phases, an **expansion** space in the flow

stream sized to create a point of **expansion** of the flow stream and in which the linear velocity of the flow stream is slowed, and at least one collection device to retain the liquid phase.

USE - The method is used for chromatography, e.g. preparative and analytical supercritical fluid chromatography (SFC) or supercritical fluid extraction for a liquid phase SFC collection system.

ADVANTAGE - The process efficiently separates liquid and **gas** phases in a flow stream upstream of a collection vessel without additional pressure schemes or **solvent** extraction imposed on the flow stream. Samples are repeatedly injected into the mobile phase flow stream and collected into large-volume containers, allowing longer unattended run times and cost-efficient sample purification and recovery.

DESCRIPTION OF DRAWING(S) - The figure shows a schematic flow diagram of a supercritical fluid chromatography system and collection system including a sample cassette.

- Thermally regulated transfer tube 12
- Back-pressure regulator 14
- Heaters 16, 18, 20
- Valve system 22
- Waste stream container 26
- Transfer tubing lines 28
- Cassette lid 30
- Discrete chambers 32
- Waste transfer line 34
- Test tube vial 36
- Liquid phase 38
- Discharge lines 40
- Pressure relief switch 42
- Molded frame 44, 46
- Butterfly latches 56
- Restrictive transfer line 72

Dwg.1/15

FS CPI EPI

FA AB; GI

MC CPI: B11-C06; B11-C09; J04-B01C

EPI: S03-E09C; S03-E13B2

TECH UPTX: 20040128

TECHNOLOGY FOCUS - INSTRUMENTATION AND TESTING - Preferred Process:

Expanding the flow stream comprises directing the stream through large bore tubing with an internal diameter sufficient to slow the linear speed of the flow stream, or comprises directing the flow stream through a chamber with an internal space sufficient to slow the linear speed of the flow stream.

Retaining the liquid phase in a collection device includes using a discrete collection container to receive the liquid phase, in which the container has an exit port for discharging waste products. The flow stream discharges from a flow line inside the collection device at an angle less than horizontal and at a tangential angle to the inner wall of the container. The process further comprises detecting the volume of the liquid phase in the collection device and stopping the flow stream filling the device when the liquid phase reaches a threshold level. The process is performed under approximate isocratic conditions.

The process further comprises injecting the samples into the process at a frequency such that a second sample injection begins elution of a sample **solute** after a first sample injection completes elution of the same **solute** within the first injection, but prior to the first sample completing an entire chromatographic process.

Determining the frequency of the sample injections comprises detecting the eluted **solutes** in the flow stream, determining time periods for elution of the **solutes** from the time of injection to the beginning of elution, determining the time periods from the start to finish of an eluted **solute** concentration peak, and automatically collecting the liquid phase containing the eluted **solutes** from

the repetitive sample injections into the collection device based on the time periods.

The process further comprises retaining the liquid phase in the collection device according to a start/stop signal from a detection device that is sent through a system controller for each of the sequential series of injections.

L116 ANSWER 2 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 2003-531794 [50] WPIX

DNC C2003-143576

TI Precipitation and retention of particle e.g. drug in carrier, by dissolving material in pressurized **gaseous** fluid or **solvent**, precipitating particles, directing into carrier mixed bed in mixed state and dispersing to produce blend.

DC B02 B03 B07

IN BOCHNIAK, D J; HORHOTA, S; KOENIG, K J; SAIM, S

PA (BOEH) BOEHRINGER INGELHEIM PHARM INC

CYC 101

PI US 2003066800 A1 20030410 (200350)* 37p B01D011-00 <--

WO 2003030871 A1 20030417 (200350) EN A61K009-16

RW: AT BE BG CH CY CZ DE DK EA EE ES FI FR GB GH GM GR IE IT KE LS LU
MC MW MZ NL OA PT SD SE SK SL SZ TR TZ UG ZM ZW

W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ DE DK
DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR
KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ OM PH PL PT
RO RU SD SE SG SI SK SL TJ TM TN TR TT TZ UA UG UZ VC VN YU ZA ZM
ZW

ADT US 2003066800 A1 Provisional US 2001-328301P 20011010, US 2002-268879
20021010; WO 2003030871 A1 WO 2002-US32303 20021010

PRAI US 2001-328301P 20011010; US 2002-268879 20021010

IC ICM A61K009-16; B01D011-00

AB US2003066800 A UPAB: 20030805

NOVELTY - Particle precipitation and retention in carrier material (CM), involves dissolving a solid or semisolid material (SSM) in a pressurized **gaseous** fluid or in a liquid **solvent**, precipitating particles, directing into a mixed bed of CM and retaining and dispersing the particles in CM to produce a blend of the SSM particles and CM. The CM in the mixed bed is maintained in a mixed state.

DETAILED DESCRIPTION - Particle precipitation and retention in carrier material (CM) comprises dissolving a solid or semisolid material (SSM) in a pressurized **gaseous** fluid or in a liquid **solvent**, to form a solution comprising a **gaseous** or liquid fluid **solvent** and a dissolved **solute** of material, precipitating SSM particles out of **gaseous** or liquid fluid solution by introducing into a region of lower pressure or into a region containing an inert **gas**, directing the introduced solution and precipitated particles onto or into a mixed bed of carrier material and retaining and dispersing the precipitated particles in the carrier material to produce a blend of the solid or semisolid material particles and carrier material, a granulation of the solid or semisolid material particles with carrier material and/or partially or fully coated with the solid or semisolid material. The carrier material in the mixed bed is maintained in a mixed state.

USE - Used for processing solution particles used in pharmaceuticals and chemical processing to obtain fine powders of drug substance. The method can be used in blending **crystallized** microparticles and nanoparticles with larger sized material and for coating of granules, pellets, non-pareils, tablets or capsules.

ADVANTAGE - The method facilitates precipitation of **solute** particle and retention and dispersion in a carrier material using pressurized **gaseous** fluids having unique properties. The method facilitates discharging and handling of the powder in downstream

processing.

Dwg.0/21

FS CPI

FA AB; DCN

MC CPI: B06-D04; B06-E05; B07-A02B; B07-D04B; B11-B; B12-M11D

TECH UPTX: 20030805

TECHNOLOGY FOCUS - PHARMACEUTICALS - Preferred Components: The precipitated particles of solid or semi-solid material (SSM) comprise microparticles or nanoparticles of SSMs. SSM Comprises physiologically active material, an encapsulating material, a moisture protection material, light protection material, **gas** protection material, diffusion barrier material or a dissolution or dispersion enhancing material. The active material comprises ipratropium bromide, tiotropium bromide, oxytropium bromide or tipranavir. The powdered carrier material comprises microparticles or nanoparticles of carrier material.

Preferred Method: The mixed bed of carrier material is maintained in a mixed state by stirring at a rate of 20-1000 (300-1000) rpm. The method produces a blend of SSM particles with carrier material. The blend of SSM particles with carrier material comprises a (non)uniform mixture of carrier material, discrete particles of SSM and carrier material having loosely adhered SSM. The coated carrier material is produced by coating several times on coated carrier material. The **gaseous** fluid solution is introduced into a region of lower pressure. The liquid solution is introduced into a region containing a pressurized **gaseous** fluid. The liquid solution is introduced into a region into which a pressurized **gaseous** fluid is subsequently introduced. The carrier material comprises lactose. The orifice through which the **gaseous** fluid solution is introduced is located within the mixed bed when the mixed bed is at rest. The SSM of active component and binder material are dissolved in the liquid **solvent** such as methanol or ethanol.

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Components: The **gaseous** fluid comprises **carbon dioxide**, nitrous oxide, trifluoromethane, ethane, ethylene, propane, sulfur hexafluoride, propylene, butane, isobutane and/or pentane. The liquid **solvent** comprises water, aliphatic alcohols, acetone, dichloromethane and/or ethyl acetate. The carrier material is in the form of powder, granulated powder, tablets capsules or caplets. The carrier material comprises a carrier, adjuvant or excipient or active material.

ABEX UPTX: 20030805

EXAMPLE - 5 g of drug substance was mixed with diatomaceous earth in a vessel. Supercritical **carbon dioxide** was supplied into the vessel at 80degreesC and drug substance was extracted and solubilized under 310 bar. The drug-laden effluent **carbon dioxide** stream was then **expanded** to a lower pressure through a 75 micro-m orifice nozzle in a mixing vessel containing 25 g of white powder of polystyrene divinyl benzene beads (particle size of 40-80 micro-m). The powder was mixed at 1000 rpm. The nozzle lip was set close to the top of the powder bed so that the drug substance precipitated as microparticles and nanoparticles were rapidly mixed with the powder. Mixing vessel temperature and pressure were 40-50degreesC and upto 1000 psig, respectively. Effluent **carbon dioxide** was passed through a 60 micro-m filter frit and was then vented. The treated powder had a yellowish, evenly distributed color, showing that the drug was uniformly distributed throughout the powder.

L116 ANSWER 3 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 2001-602608 [68] WPIX

DNC C2001-178498

TI Processing **solute** for, e.g. **recrystallization** of dissolved material from solution, employs **solvent**

expansion-contraction.

DC B07
 IN BOCHNIAK, D J; HORHOTA, S; SAIM, S
 PA (BOEH) BOEHRINGER INGELHEIM PHARM INC; (BOCH-I) BOCHNIAK D J; (HORH-I) HORHOTA S; (SAIM-I) SAIM S
 CYC 36
 PI WO 2001066215 A1 20010913 (200168) * EN 48p B01D011-02 <--
 RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE TR
 W: AU BR CA CN CZ HU IL IN JP KR MX NZ PL RU TR US ZA
 AU 2001034659 A 20010917 (200204) B01D011-02 <--
 US 2001055561 A1 20011227 (200206) B01D011-00 <--
 EP 1263516 A1 20021211 (200301) EN B01D011-02 <--
 R: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE TR
 BR 2001008912 A 20021224 (200309) B01D011-02 <--
 KR 2002077523 A 20021011 (200314) B01D011-02 <--
 CZ 2002003273 A3 20030514 (200337) B01D011-02 <--
 HU 2003000065 A2 20030528 (200341) B01D011-02 <--
 CN 1411389 A 20030416 (200345) B01D011-02 <--
 ZA 2002006943 A 20030625 (200348) 53p B01D000-00 <--
 JP 2003525731 W 20030902 (200358) 39p B01J019-00
 MX 2002008331 A1 20030101 (200373) B01D011-02 <--
 ADT WO 2001066215 A1 WO 2001-US3019 20010130; AU 2001034659 A AU 2001-34659 20010130; US 2001055561 A1 Provisional US 2000-186888P 20000303, US 2001-774232 20010130; EP 1263516 A1 EP 2001-906792 20010130, WO 2001-US3019 20010130; BR 2001008912 A BR 2001-8912 20010130, WO 2001-US3019 20010130; KR 2002077523 A KR 2002-711573 20020903; CZ 2002003273 A3 WO 2001-US3019 20010130, CZ 2002-3273 20010130; HU 2003000065 A2 WO 2001-US3019 20010130, HU 2003-65 20010130; CN 1411389 A CN 2001-806012 20010130; ZA 2002006943 A ZA 2002-6943 20020829; JP 2003525731 W JP 2001-564861 20010130, WO 2001-US3019 20010130; MX 2002008331 A1 WO 2001-US3019 20010130, MX 2002-8331 20020827
 FDT AU 2001034659 A Based on WO 2001066215; EP 1263516 A1 Based on WO 2001066215; BR 2001008912 A Based on WO 2001066215; CZ 2002003273 A3 Based on WO 2001066215; HU 2003000065 A2 Based on WO 2001066215; JP 2003525731 W Based on WO 2001066215; MX 2002008331 A1 Based on WO 2001066215
 PRAI US 2000-186888P 20000303; US 2001-774232 20010130
 IC ICM B01D000-00; B01D011-00; B01D011-02; B01J019-00
 ICS B01D009-00; B01D009-02; B09B003-00
 AB WO 2001066215 A UPAB: 20011121
 NOVELTY - A **solute** is processed by dissolving it in a **solvent**; dissolving a **gas** in the solution; causing the solution to **expand** through a filter; causing the **gas** to be dissolved to a concentration such that the solution **expands**; retaining precipitated **solute** on a filter; reducing the pressure in the solution to expel the **gas**; and optionally adding more **solute** to the resultant solution.
 DETAILED DESCRIPTION - Processing a **solute** comprises
 (a) dissolving at least a portion of the **solute** in a liquid **solvent** that has an affinity for the solubilization of the **solute**;
 (b) dissolving a **gas** in the solution;
 (c) causing the solution to **expand** through a filter that can retain unsolubilized **solute** particles;
 (d) causing the **gas** to be dissolved to a concentration such that the solution **expands** until it loses its affinity for the solubilization of the **solute** and the **solute** precipitates;
 (e) retaining precipitated **solute** on a filter which is the same as or different from the filter used in step (c);
 (f) reducing the pressure in the solution such that the **gas** is expelled, providing a resultant solution having an affinity for the solubilization of the **solute**; and

(g) optionally adding more **solute** to the resultant solution.

USE - For processing a **solute** for, e.g. **recrystallization** of a dissolved material from a solution, extraction of material from a composition, coating of a material on a substrate, impregnating a material into a matrix, removal of contaminants from an article, or chemical reactions (claimed).

ADVANTAGE - The inventive process employs minimum consumption of the organic **solvent** and **gas**, and reduced operating and capital costs. It operates at low temperatures and pressures such that environmental friendliness is enhanced. The **solvent** can easily be adjusted and can be reused for extraction, and little or no extract is typically lost.

Dwg.0/10

FS

CPI

FA

AB; DCN

MC

CPI: B05-C04; B11-C01

TECH

UPTX: 20011121

TECHNOLOGY FOCUS - CHEMICAL ENGINEERING - Preferred Method: Steps (a)-(f) are repeated at least one more time, or steps (a)-(g) are repeated at least three times.

TECHNOLOGY FOCUS - PHARMACEUTICALS - Preferred Material: The **solute** is a pharmaceutical drug substance, an impurity, or an intermediate product in the synthesis of a pharmaceutical drug substance.

TECHNOLOGY FOCUS - INORGANIC CHEMISTRY - Preferred Material: The **gas** is **carbon dioxide**.

L116 ANSWER 4 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 2000-387648 [33] WPIX

DNC C2000-117665

TI Continuous harvesting of particles from organic solution-laden near critical and supercritical fluids uses filter consisting of thin membrane supported on sintered stainless steel tube.

DC B07

IN BOCHNIAK, D J; RAJEWSKI, R A; SUBRAMANIAM, B

PA (UNIV) UNIV KANSAS

CYC 87

PI WO 2000029096 A1 20000525 (200033)* EN 30p B01D061-00 <--
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW NL
OA PT SD SE SL SZ UG ZW

W: AE AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB
GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU
LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR
TT UA UG UZ VN YU ZA ZW

AU 9958183 A 20000605 (200042) B01D061-00 <--
US 6113795 A 20000905 (200044) B01D061-00 <--
EP 1133345 A1 20010919 (200155) EN B01D061-00 <--
R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK NL PT
RO SE SI

AU 753461 B 20021017 (200280) B01D061-00 <--

ADT WO 2000029096 A1 WO 1999-US20651 19990909; AU 9958183 A AU 1999-58183
19990909; US 6113795 A US 1998-193660 19981117; EP 1133345 A1 EP
1999-945612 19990909, WO 1999-US20651 19990909; AU 753461 B AU 1999-58183
19990909

FDT AU 9958183 A Based on WO 2000029096; EP 1133345 A1 Based on WO 2000029096;
AU 753461 B Previous Publ. AU 9958183, Based on WO 2000029096

PRAI US 1998-193660 19981117

IC ICM B01D061-00

AB WO 200029096 A UPAB: 20000712

NOVELTY - A feed stream is fed into the separator, containing a porous layer (56), at a pressure of 0.5 to 1 Pc. The feed stream consists of the

particles and a mixture including a **solvent** and an **antisolvent** for the particles. The feed stream contacts the porous layer (56). At least some of the mixture passes through the layer and at least some of the particles are separated by it.

DETAILED DESCRIPTION - The **antisolvent** may be **carbon dioxide**, propane, butane, isobutane, nitrous oxide, sulfur hexafluoride, trifluoromethane, methane, hydrogen, or mixtures of these. The feed stream is introduced under supercritical conditions for the mixture. The **solvent** is miscible with the **antisolvent** at this pressure. The **solvent** is an organic **solvent**. The separator consists of two porous layers, the first (70) being a membrane of titanium dioxide with a thickness of 0.5 to 40 microns, and the second (72) a porous sintered stainless steel. The feed stream is prepared prior to being introduced to the separator by contacting the **antisolvent** with a dispersion including a **solute** dissolved in the **solvent** so that at least some of the **solute** precipitates out of the dispersion to form the particles.

USE - For continuously harvesting micro- and nano-particles from near-critical or supercritical fluids. In specific examples, the particles are pharmaceuticals, e.g. a cancer treating agent, a pharmaceutical for use in intravenous injections or particles for use in inhalation therapy.

ADVANTAGE - The method provides an increased rate of production and harvesting. No chemical reactions take place in the process resulting in particles which are the same chemically as the drug used to form the dispersion.

DESCRIPTION OF DRAWING(S) - The figure shows schematically the high pressure filter.
porous layer 56

porous membrane 70

sintered stainless steel tube 72

Dwg.2/6

FS CPI

FA AB; GI; DCN

MC CPI: B05-C03; B05-C07; B05-C08; B10-H02B; B10-J02; B11-B; B12-M11E;
B14-H01

L116 ANSWER 5 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 2000-183881 [17] WPIX

DNC C2000-057847

TI Production of spherical particles, especially of e.g. pharmaceuticals, comprises **crystallization** on spherical seed **crystals**.

DC B04

IN HEFFELS, S; NICOLAOU, I; SCHUNK, W

PA (AVET) AVENTIS RES & TECHNOLOGIES GMBH & CO KG; (AXIV-N) AXIVA GMBH

CYC 25

PI DE 19834876 A1 20000203 (200017)* 6p B01D009-02 <--

WO 2000007685 A1 20000217 (200017) DE B01D009-00 <--

RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE

W: BR BY IN MX PL RU US

ADT DE 19834876 A1 DE 1998-19834876 19980801; WO 2000007685 A1 WO 1999-EP4787 19990708

PRAI DE 1998-19834876 19980801

IC ICM B01D009-00; B01D009-02

ICS A61K009-10; A61K009-16

AB DE 19834876 A UPAB: 20000405

NOVELTY - Process (A) for producing particles comprises **crystallization** using spherical seed **crystals**.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for the following:

(1) a process (B) for producing spherical seed **crystals**, comprising spray drying a solution of the substance to be **crystallized**;

(2) a process (C) for producing spherical particles, comprising dispersing the substance to be **crystallized** in an immiscible organic **solvent** and **crystallizing** the substance in the resulting droplets;

(3) particles obtainable by processes (A), (B) or (C).

USE - The process is especially useful for producing spherical particles of pharmaceuticals, e.g. cefotaxime disodium or piratenide, or special chemicals, e.g. phenylhydrazines, either by **crystallization** from melts, solutions, **gases** or supercritical media or by precipitation or reactive **crystallization**, optionally where the particles comprise several shell-like layers, at least two of which have a different composition.

ADVANTAGE - Spherical **crystals**, which have good flow properties, can be produced without the need for large-scale extractive **crystallization** in droplets dispersed in an immiscible **solvent**. The spherical seed **crystals** can be produced by simple spray drying.

Dwg.0/3

FS CPI

FA AB; DCN

MC CPI: B06-F03; B10-A19; B11-B

ABEX UPTX: 20000405

EXAMPLE - A 15% aqueous solution of cefodizime disodium (I) was spray dried with nitrogen to produce spherical particles with a size of 9 μm . An aqueous solution of (I) was **crystallized** by dilution with ethanol in the presence of 1 weight% (based on **solute**) of the spherical particles. The product was filtered and dried to give largely spherical **crystals** with an average particle size of 16 μm .

L116 ANSWER 6 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 1994-349384 [43] WPIX

DNC C1994-159105

TI Gas-evolution separation of a **solute** from solution in a **solvent** - by dissolving a **gas** forming material in the soln and adjusting pressure and temperature to provide a three phase separation.

DC D15 E33 J01 M25

IN FERRAMOSCA, A C; LURIE, W; SLOAN, J C

PA (PARH-N) PARHELION INC

CYC 1

PI US 5360554 A 19941101 (199443)* EN 17p C02F001-22

ADT US 5360554 A US 1994-192725 19940207

PRAI US 1994-192725 19940207

IC ICM C02F001-22

ICS B01D009-04

AB US 5360554 A UPAB: 19941216

Solute is separated from a solution in a **solvent** by dissolving a **gas** forming material (fr.40) in the solution (16) lowering the temperature of the solution placing the solution in a pressure vessel (52) and increasing its pressure to a nominal high value from which it is released to allow the major portion of the **gas** forming material and a minor portion of the **solvent** to form vapours that undergo a Joule-Thompson free **expansion** into a closed second vessel (40) at a low pressure to obtain three phases of resultant materials that each have a temperature approximating the triple point temperature of the solution. The three phases comprise a **gas** phase product containing the **gas** forming material and vapours of the **solvent**, a liquid phase product with **solute** concentration greater than the initial concentrate of the **solute/solvent** soln starting material, and a solid phase form of the **solvent**. One of the phases is collected as product of the process, a portion of the **gas** phase product is collected from reuse (at 26) in the process, a portion of the **gas** phase product is

collected from reuse (at 26) in the process, and one of the phases is recycled into an earlier stage of the process via a heat exchanger (38) that heat exchanges a relatively cold resultant material with a relatively warm **solute/solvent** solution. Also claimed is the process in which the liquid phase product with increased **solute** concentration is recycled (86,96) to the initial **solute/solvent** soln to adjust its **solute** concentration. Heat exchangers (46,48) are used to control the temperature of the solution prior to pressurising in the pressure vessel, a jet eductor (24) is used to collect the **gas** phase product for re-use, and the liquid product is either recycled via a heat exchanger, or separated into a constant recycle quantity and a remaining 'Blow-Down' quantity comprising total **solute** of the starting material and unsolidified remaining **solvent** in proportion up to the eutectic proportion of the starting **solute/solvent** solution.

USE - In converting sea or brackish water into potable water, recovering metals such as magnesium from sea water etc. de mineralising fresh water to make carbonated beverages, recovering **solutes** or **solvents** from industrial process **solvents**, or cleaning up polluted bodies of water.

ADVANTAGE - Only the vapour products are **expanded** into the second vessel reducing energy expenditure yet producing substantial quantities of solid **solvent**.

Dwg.2/2

FS CPI

FA AB; GI; DCN

MC CPI: D04-A01F; E11-Q01; E31-N05C; E34-B; J01-C; J01-D; M25-F; M25-G16

L116 ANSWER 7 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 1991-261186 [36] WPIX

CR 1991-347857 [48]; 1993-183877 [23]; 1993-338219 [43]

DNC C1991-113366

TI Supercritical fluid extraction with independent control of conditions - comprises which system, maintains **solvent**, flow, temperature and pressure using a variable **expansion** nozzle.

DC J01

IN DRYDEN, P C; ENGEL, S J; FRANK, L R; WURM, C M

PA (HEWP) HEWLETT-PACKARD CO

CYC 5

PI EP 444299 A 19910904 (199136)*

R: DE FR GB

US 5133859 A 19920728 (199233) 13p B01D015-08 <--

JP 04222602 A 19920812 (199239) 11p B01D011-00 <--

EP 444299 B1 19950222 (199512) EN 15p B01D011-02 <--

R: DE FR GB

DE 69017190 E 19950330 (199518) B01D011-02 <--

JP 3207866 B2 20010910 (200155) 10p B01D011-00 <--

ADT EP 444299 A EP 1990-125072 19901221; US 5133859 A US 1990-487693 19900302;

JP 04222602 A JP 1991-59543 19910301; EP 444299 B1 EP 1990-125072

19901221; DE 69017190 E DE 1990-617190 19901221, EP 1990-125072 19901221;

JP 3207866 B2 JP 1991-59543 19910301

FDT DE 69017190 E Based on EP 444299; JP 3207866 B2 Previous Publ. JP 04222602

PRAI US 1990-487693 19900302

REP EP 206739; EP 275933; EP 296145; JP 06214885; JP 62148855

IC ICM B01D011-00; B01D011-02; B01D015-08

ICS G01N001-10

AB EP 444299 A UPAB: 20010927

A method for the supercritical extraction of one component of a sample uses a flow system having control equipment for the pressure and temperature of the extraction medium. The control equipment operates in conjunction with a variable flow restrictor nozzle to control the condition of the fluid flowing through the sample chamber (. Pressurised extraction fluid is supplied from a cylinder via a pump (with a pressure regulator to the

sample chamber.

The fluid is exhausted via the **expansion** nozzle (. The pump injects the fluid into the system at a controlled, predetermined, flowrate and the system pressure is controlled by setting the variable nozzle as appropriate. The equipment also controls to a predetermined extraction time.

Pref. additional features include a bypass to allow the **solvent** to be routed away from the sample chamber, and a nozzle and trap system for collecting a sample of the material after the extraction. Rinse **solvent** may be passed through the sample trap after collection to remove selected fractions for analysis or further treatment. A sample of **solvent** containing the extracted **solute** may also be collected after it has left the extraction chamber.

USE/ADVANTAGE - Improved method of supercritical fluid extraction. Control system allows independent selection and control of pressure and temperature of extraction medium (i.e., its **solvent** power). Aopts. can use any convenient vessel as a sample container. @(12pp Dwg.No.0/3)

FS CPI

FA AB

MC CPI: J01-C01

ABEQ US 5133859 A UPAB: 19930928

Apts. for components extn. from a sample, specifically by **gas** or liq. chromatography or supercritical fluid chromatography, comprises a **gas** liquefaction pump controlled to have regulated output pressure for supplying a chamber contg. the sample via a variable orifice nozzle controlled by a pressure transducer, so that the **gas** pressure in the chamber is at a set point value.

Pref. the **gas** exits the chamber via a trap contg. porous granular material, which is inert, chemically active or adsorbent.

USE - Chromatography using liq. **CO2** as sample component extn. **solvent**.

ABEQ EP 444299 B UPAB: 19950328

Apparatus for the extraction of components from a sample comprising: (a) one or more sources of **solvent** fluid (100); (b) one or more extraction **solvent** fluid input ports (101), (c) a controllable high pressure pump (202); (d) a pressure transducer (240a) to measure the pressure of the fluid delivered by the high pressure pump (202); (e) a flow transducer (226); (f) an **expansion** nozzle section having a variable and controllable flow restriction (108); (g) a control apparatus for controlling independently the variable flow restriction and the high pressure pump (202) so as to achieve and to maintain a set point pressure and a set point flow rate; (h) an extraction chamber flow system (209) comprising an extraction chamber (210) for retaining the sample in the flow stream of the fluid and a sample input module for containing the sample in the extraction chamber (210); (i) a bypass flow system (207) which routes fluid flow around said extraction chamber section (k) means (213) for merging the bypass flow system (207) and extraction chamber flow system (209) together; (l) sample collection means for separating the extracting **solvent** fluid from components from said sample, comprising at least one nozzle (216) and trap (218) subassembly, said nozzle forming part of said **expansion** nozzle section; and (m) at least one sample collection vessel (236) for collecting the components from said sample.

Dwg.3/3

L116 ANSWER 8 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 1981-28359D [16] WPIX

TI Purificn. of **crystalline** solid from solution - by pressurising, releasing pressure and discharging **solvent** by pressure of **gas** (J5 19.8.75).

DC J01

PA (KOBM) KOBE STEEL LTD

CYC 1

PI JP 56012161 B 19810319 (198116) *

JP 50104771 A 19750819 (198116)

PRAI JP 1974-11328 19740125

IC B01D009-02

AB JP 81012161 B UPAB: 19930915

Method comprises pressurising solution in a pressure vessel to solidify the **solute**, releasing the pressure rapidly to such a level as to re-dissolve a part of the solid, and discharging the **solvent** by pressure of **gas** which was initially charged in the vessel.

Used for purifying **crystalline** solid from solution (J50104771).

FS CPI

FA AB

MC CPI: J01-B

=>